BRUKER

Bruker AXS

APEX2 Release 2010

User Manual

Part Number: DOC-M86-E02078

Publication Date: 13 May 2010

think forward

X-ray Diffraction

This document covers the APEX2 Suite of programs.

References to this document should be shown as DOC-M86-EXX078 APEX2 User Manual.

© 2010 Bruker AXS Inc., 5465 East Cheryl Parkway, Madison, WI 53711. All world rights reserved.

Notice

The information in this publication is provided for reference only. All information contained in this publication is believed to be correct and complete. Bruker AXS Inc. shall not be liable for errors contained herein, nor for incidental or consequential damages in conjunction with the furnishing, performance, or use of this material. All product specifications, as well as the information contained in this publication, are subject to change without notice.

This publication may contain or reference information and products protected by copyrights or patents and does not convey any license under the patent rights of Bruker AXS Inc. nor the rights of others. Bruker AXS Inc. does not assume any liabilities arising out of any infringements of patents or other rights of third parties. Bruker AXS Inc. makes no warranty of any kind with regard to this material, including but not limited to the implied warranties of merchantability and fitness for a particular purpose.

No part of this publication may be stored in a retrieval system, transmitted, or reproduced in any way, including but not limited to photocopy, photography, magnetic, or other record without prior written permission of Bruker AXS Inc.

Address comments to:

Technical Publications Department
Bruker AXS Inc.
5465 East Cheryl Parkway
Madison, Wisconsin 53711-5373
USA

All trademarks and registered trademarks are the sole property of their respective owners.

Printed in the U.S.A.

Revision History

Revision	Date	Changes
0	February 2005	Initial release.
1	June 2006	Revised and expanded for APEX2 Version 2.
2	13 May 2010	Revised and expanded.

Bruker Corporation

40 Manning Road
Billerica, MA USA

Phone +1 (978) 663-3660
Fax: +1 (978) 667-5993

E-mail: info@bruker-biosciences.com
www.bruker-biosciences.com

Bruker AXS Inc.

5465 East Cheryl Parkway
Madison, WI 53711-5373 USA

Phone +1 (800) 234-XRAY [9729]
Fax +1 (608) 276-3006

E-mail: info@bruker-axs.com
www.bruker-axs.com

MCNA
ISO 9001:2008
Certified
Management Certification
of North America
ANAB
ACCREDITED

Table of Contents

1 About this User Manual 1-1

1.1 APEX2 Software for Chemical Crystallography 1-1

1.2 The Ylid Crystal 1-2

1.3 Terms and Conventions 1-3

1.3.1 Typographical Conventions 1-3

1.3.2 Equivalent Terms 1-3

1.4 Referenced Documentation 1-5

1.5 X-ray Safety 1-6

1.6 Help and Technical Support 1-7

1.6.1 The “What’s This?” Help Function 1-7

1.6.2 Technical Support from Bruker AXS Service Centers 1-7

2 APEX2 Software Overview 2-1

2.1 Introduction to Client and Server Functions 2-2

2.1.1 Online and Offline Operation 2-2

2.2 Server Programs 2-3

2.2.1 The Bruker Instrument Service (BIS) 2-3

2.3 Client Programs 2-4

2.3.1 APEX2 GUI and Modules 2-4

2.3.2 The Bruker Configuration Program (BCP) 2-4

2.3.3 APEX2Server 2-5

2.4 The Sample Database 2-6

2.5 License Considerations 2-6

2.6 The Main Window 2-7

2.6.1 The Title Bar 2-8

2.6.2 The Menu Bar 2-8

2.6.3 The Tool Icon Bar 2-10

2.6.4 The Task Bar 2-11

2.6.5 The Task Display Area 2-14

3 Startup and Shutdown 3-1

3.1 User Access 3-1

3.2 Startup 3-2

3.2.1 Start BIS 3-2
3.2.2 Start APEX2 3-2
3.2.3 Log In 3-2
3.2.4 Connect to BIS 3-2

3.3 Shutdown 3-3

3.3.1 Shut Down APEX2 3-3
3.3.2 Shut Down BIS 3-3

4 Crystal Centering and Screening 4-1

4.1 Centering the Crystal 4-1

4.1.1 Start the Center Crystal Module 4-2
4.1.2 Mount the Goniometer Head 4-2
4.1.3 Center the Crystal on the KAPPA Series II Goniometer 4-4
4.1.4 Center the Crystal on the D85 Goniometer 4-10

4.2 Checking Crystal Quality 4-15

4.2.1 Start the Simple Scans Module 4-15
4.2.2 Set up the Scan 4-15
4.2.3 Use the Resolution Circle Tool 4-17
4.2.4 Use the Line Tool 4-18
4.2.5 Plot a Rocking Curve 4-19
4.2.6 View Peak Profiles in 3D 4-20
4.2.7 Examples of Poor-Quality Crystals 4-21

5 Data Collection 5-1

5.1 Create a New Sample 5-2

5.2 Describe the Sample with the Describe Module 5-2

5.3 Determining the Unit Cell 5-3

5.3.1 Start the Determine Unit Cell Module 5-4
5.3.2 Determine the Unit Cell in Automatic Mode 5-4
5.3.3 Manual Mode 5-5

5.4 Determining the Data Collection Strategy 5-14

5.4.1 Using the Collect-Style Data Collection Strategy Module 5-15
5.4.2 Using the Data Collection Strategy Module 5-20

5.5 Run the Experiment 5-26

5.5.1 User-Defined Runs and Settings 5-28
5.5.2 Exiting During Data Collection 5-28
5.5.3 Abort the Experiment if Necessary 5-28

6 Data Integration and Scaling 6-1

6.1 Start the Integrate Images Module 6-2

6.2 Set the Resolution Limit 6-2

6.3 Select Runs for Integration 6-3

6.3.1 Browse for Runs 6-3
6.3.2 Import Runs from Experiment 6-4
6.3.3 Modifying Integration Run Lists 6-4

6.4 Set the Refinement Options 6-5

6.4.1 Refinement Options Controls 6-6

6.5 Set the Integration Options 6-7

6.5.1 Integration Options Controls 6-8

6.6 Start the Integration 6-10

6.6.1 Monitoring Integration Progress with SaintChart 6-10

6.7 Examine the Integration Results 6-13

6.7.1 Overall Rsym and Coverage Statistics 6-13
6.7.2 Unconstrained Unit Cell Refinement 6-14

6.8 Scaling Data 6-15

6.8.1 Start the Scale Module 6-16
6.8.2 Set Up the Scale Operation 6-16
6.8.3 Refine the Parameters 6-18
6.8.4 Refine the Error Model 6-20
6.8.5 Examine the Diagnostic Plots 6-21
6.8.6 Close the Scale Module 6-23

7 Examining Data 7-1

7.1 Using the Space Groups and Statistics Module 7-2

7.1.1 Start the Space Groups and Statistics Module 7-2
7.1.2 Set Up the Determination Parameters 7-2
7.1.3 Examine the Lattice Exceptions 7-3
7.1.4 Choose a Space Group 7-4
7.1.5 Examine the Reflection Statistics 7-5
7.1.6 Check the Unit Cell Contents 7-7
7.1.7 Set Up the Output File 7-8
7.1.8 Examine Cell Information 7-9
7.1.9 Examine Diagnostics 7-10
7.1.10 Exit the Space Groups and Statistics Module 7-10

7.2 Determining the Space Group with XPREP 7-11

7.2.1 Determine the Space Group 7-12
7.2.2 Examine Reflection Statistics 7-14
7.2.3 Prepare an Output File 7-16
7.2.4 Quit XPREP 7-17

7.3 Create Simulated Precession Images 7-18

8 Structure Solution and Refinement. 8-1

8.1 Using AUTOSTRUCTURE 8-1
8.1.1 Before you Begin 8-1
8.1.2 Solving the Structure 8-2
8.1.3 Automatically Solving and Refining the Structure 8-3

8.2 Solving the Structure with the Structure Solution Module. 8-4
8.2.1 Direct Methods Options 8-6
8.2.2 Dual-Space Methods Options 8-6
8.2.3 Patterson Methods Options 8-6

8.3 Refining the Structure with the Structure Refinement Module 8-7
8.3.1 Refining the Structure in XSHELL 8-9
8.3.2 Labelling the Atoms 8-12
8.3.3 Refining the Model. 8-13
8.3.4 Refining for Publication with XSHELL 8-21
8.3.5 Creating a Publication-Quality Image 8-22

9 Using the Report Generation Module 9-1

9.1 Preparing for Report Generation 9-1

9.2 Using the Module 9-2
9.2.1 Starting the Module 9-2
9.2.2 Choosing a Template File 9-3

9.3 Creating an HTML-Formatted Report for Online or Print Distribution 9-4
9.3.1 Saving the HTML-Formatted Report 9-4

9.4 Creating a Report for Submission to Acta Crystallographica 9-5
9.4.1 Saving the CIF-Formatted Report 9-5

9.5 Adding Additional Information to the Report 9-6
9.5.1 Adding Information from a .CIF File. 9-7
9.5.2 Adding the Run List 9-8
9.5.3 Adding Additional Information 9-9

9.6 Report Sections 9-11
9.6.1 “Report” Tab. 9-11
9.6.2 “Atoms” Tab 9-12
9.6.3 “Bond Lengths” Tab 9-13
9.6.4 Bond Angles 9-14
9.6.5 “Torsion Angles” Tab 9-15
9.6.6 “Miscellaneous” Tab. 9-16

10 Appendices 10-1

10.1 Using BCP with APEX2 10-2
10.1.1 Start BCP 10-2
10.1.2 Select a Connection Mode 10-2
10.1.3 Parameter Menus 10-2
10.1.4 Change the Detector's Beam Center 10-3
10.1.5 Collect a Dark Current Correction 10-5

10.2 Set Default Orientation Matrix Scans in the bn-config.py File 10-6

10.3 Face Indexing with the Crystal Faces Module 10-7
10.3.1 Starting the Crystal Faces Module 10-7
10.3.2 Collecting a Video Zip Stream 10-7
10.3.3 The Crystal Faces Module Interface 10-9
10.3.4 Specifying Crystal Faces 10-11
10.3.5 Editing the Face List 10-13
10.3.6 Scaling with Face Indices 10-14

10.4 The Reciprocal Lattice Viewer Module 10-15
10.4.1 Start the Reciprocal Lattice Viewer Module 10-15
10.4.2 Views and Tools 10-15
10.4.3 Defining Groups 10-28
10.4.4 Measuring Distances and Angles 10-30
10.4.5 Writing a .p4p File 10-30

10.5 Using CELL_NOW 10-31
10.5.1 Running CELL_NOW on a List of Reflections 10-31
10.5.2 CELL_NOW Output 10-36

10.6 Configuring the Suite 10-37
10.6.1 The bn-config Configuration File 10-37
10.6.2 Global, User, and Sample-specific Variables 10-38
10.6.3 Configuration File Examples 10-39
10.6.4 Functionality Groups in the Configuration File 10-41
10.6.5 Configuration File Variables 10-42

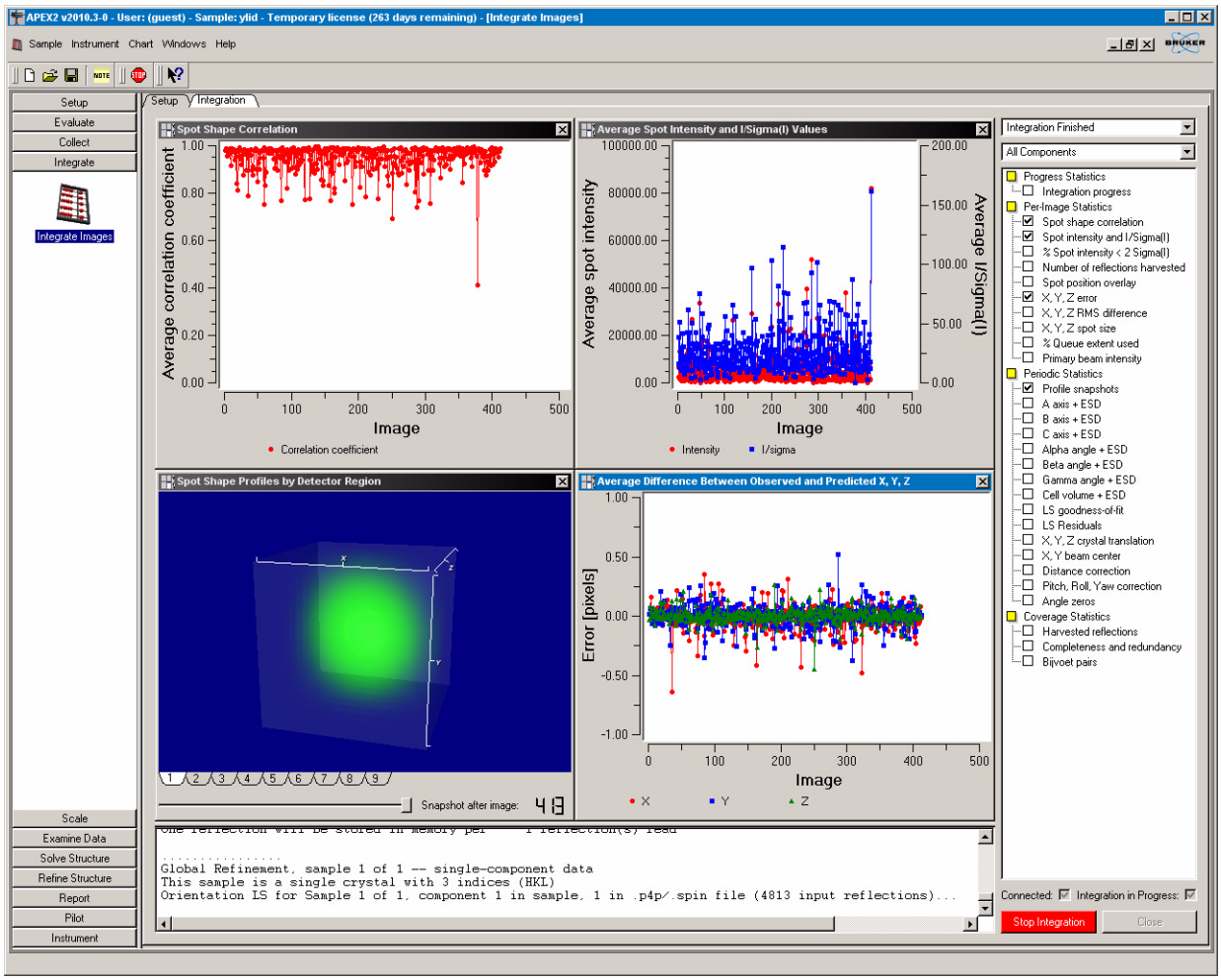
Index Index-1

This page intentionally left blank.

1 About this User Manual

1.1 APEX2 Software for Chemical Crystallography

Figure 1.1 — APEX2 software (Integrate Images module shown)



APEX2 is Bruker's comprehensive software suite for single-crystal X-ray diffraction. APEX2, combined with Bruker's X-ray diffractometer hardware, provides the tools for complete small-molecule structure determination. The software provides the highest-quality data: from acquisition, collection, integration and scaling, through structure solution and refinement, through report generation.

APEX2 redefines user interaction with the crystallographic experiment. The user interface guides the user through the complete experiment with minimal user input and maximal graphical feedback. APEX2 lets the user focus on the structure determination at hand and does not require any knowledge of instrument geometry or data collection strategies. APEX2 is easy to use for the novice, but has all of the features required by expert crystallographers.

The software features a client/server architecture that provides for remote control of—and access to—the instrumentation.

Optional software modules for APEX2 include XRD^2 Eval for powder diffraction pattern integration, and AUTOSTRUCTURE for completely-automated structure solution.

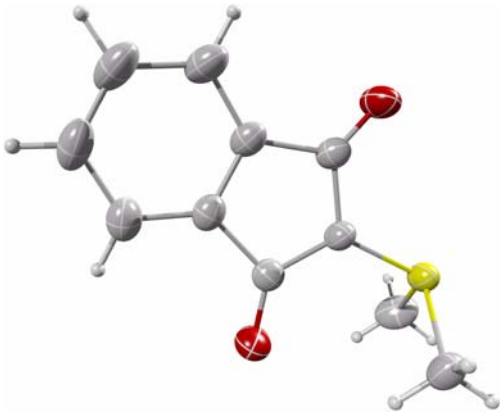
1.2 The Ylid Crystal

The compound used was 2-dimethylsulfuranylidene-1,3-indanedione (abbreviated “ylid”):

$C_{11}H_{10}O_2S$

More information on the ylid crystal is available in the article “Polymorphism and History of 2-Dimethylsufuranylidene-1,3-indanedione (YLID)”, Ilia A. Guzei, Galina A. Bikzhanova, Lara C. Spencer, Tatiana V. Timofeeva, Tiffany L. Kinnibrugh and Charles F. Campana. *Cryst. Growth Des.*, **2008**, 8 (7), pp. 2411–2418, DOI: 10.1021/cg701260p.

Figure 1.2 — Ylid crystal structure



1.3 Terms and Conventions

1.3.1 Typographical Conventions

Table 1.1 shows typographical conventions used to help you quickly locate and identify information in this document.

Table 1.1 — Typographical conventions

Convention	Usage
boldface	Software user interface controls (such as icons, menu items, and buttons) to be selected as part of the current procedure.
italics	New terms and words requiring emphasis.
`monospace`	Information read from or entered into a field or command prompt.
>	Navigation through a hierarchical menu. For example, “Choose **Start > Programs > Bruker AXS > BIS**” describes navigating Windows’ menus from **Start** to **Programs** to **Bruker AXS** to **BIS**.
[square brackets]	Keyboard input.

1.3.2 Equivalent Terms

Frame/Image

In 2-dimensional X-ray diffraction, the terms *frame* and *image* refer to the same 2-dimensional view of the diffraction pattern.

Greek and Roman Text

This document uses scientific terminology that may be rendered in Greek text. However, this document follows a convention of using Roman text to the greatest extent possible.

Table 1.2 — Greek and Roman Text

Greek	Roman
2θ	2theta
ω	omega
φ	phi
χ	chi
κ	kappa

Notices: Danger, Caution, and Note

This document contains notices that must be observed to ensure personal safety, as well as to protect the product and connected equipment. These notices are highlighted as follows according to the level of danger.

DANGER

The word “DANGER” alerts you to an immediate or potential hazard that can result in death, severe personal injury, or substantial property damage.

CAUTION

The word “CAUTION” alerts you to a potential practice or condition that could result in minor personal injury or damage to the product or property.

NOTE: The word “NOTE” in bold capital letters draws your attention to particularly important information on the product or handling of the product, or to a particular part of the product documentation.

1.4 Referenced Documentation

The following Table contains a list of documentation referenced by this document. It is necessary to have this additional documentation available as you work with this document. In the documents' part numbers, a variable revision number is indicated by a letter "X". Always use the most current revisions available.

All of the documents shown in the Table may be found at Bruker AXS' comprehensive support site www.brukersupport.com, or on the Online Documentation CD-ROM that accompanies the shipment.

Table 1.3 — Referenced documentation

Part Number	Title
DOC-269-0159XX	SHELXTL Software Reference Manual
DOC-269-0175XX	SAINT Software Reference Manual
DOC-269-0228XX	VIDEO Installation Notes
DOC-M86-EXX010	COSMO Installation Notes
DOC-M86-EXX024	D8 and PLATFORM Microscope Focus and Sample Alignment Procedure
DOC-M86-EXX043	BCP and BIS Installation Notes
DOC-M86-EXX045	RLATT User Manual
DOC-M86-EXX087	APEX2 and PROTEUM2 Installation Notes
DOC-M86-EXX092	User Manager User Manual
DOC-M86-EXX112	COSMATIC Fixed-Chi Geometry Hardware Profile Procedure
DOC-M86-EXX113	COSMATIC Kappa Geometry Hardware Profile Procedure
DOC-M88-EXX099	License Manager User Manual

1.5 X-ray Safety

DANGER

X-ray equipment produces potentially harmful radiation and can be dangerous to anyone in the equipment's vicinity unless safety precautions are completely understood and implemented. All persons designated to operate or perform maintenance on this instrument must be fully trained on the nature of radiation, X-ray generating equipment, and radiation safety. All users of the X-ray equipment are required to accurately monitor their exposure to X-rays by proper use of X-ray dosimeters.

For safety issues related to operation and maintenance of your particular X-ray generator, diffractometer, and shield enclosure, please refer to the manufacturer's operation manuals or to your Radiation Safety Officer. The user is responsible for compliance with local safety regulations.

1.6 Help and Technical Support

1.6.1 The “What’s This?” Help Function

Context-based help is available at any time within the program by simply clicking the “What’s This?” help icon:

Figure 1.3 — “What’s This?” help icon

After clicking the icon, click on any window or field to obtain detailed help in a pop-up window.

The “What’s This?” help function can also be invoked at any time by pressing [Shift]+[F1].

1.6.2 Technical Support from Bruker AXS Service Centers

You are invited to contact Bruker AXS whenever there are problems or questions related to the system. Before contacting Bruker AXS, please:

- If you suspect a software problem, determine the system’s software version.
- Record any error messages that appear.
- If possible, determine conditions and steps that recreate the problem.

CAUTION

Failure to refer instrument servicing to qualified Bruker personnel may result in injury or property damage!

North American Service Center Contact Information

Table 1.4 — Bruker AXS North American Service Center contact information

Bruker AXS North American Service Center	
Address:	Bruker AXS Inc. Customer Support 5465 East Cheryl Parkway Madison, WI 53711-5373 USA
Toll-free telephone:	1 (800) 234-XRAY [9729]
Direct line:	1 (608) 276-3087
Fax:	1 (608) 276-3015
E-mail:	Customer.Service@bruker-axs.com
Web:	http://www.bruker-axs.com

Outside North America

Outside North America, contact your local Bruker AXS Service Center.

This page intentionally left blank.

2 APEX2 Software Overview

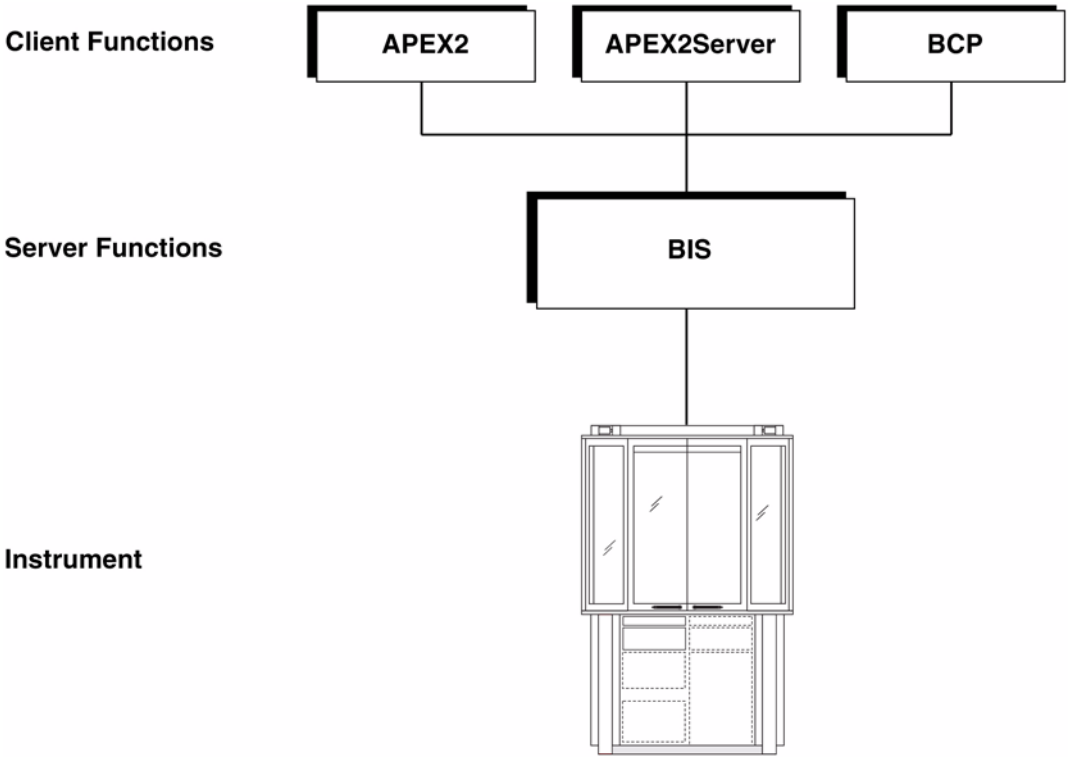
From a software and operational viewpoint, the APEX2 software controls all operations—from crystal screening to report generation—for a typical crystallography study.

This is a complete departure from the older, functionally-separate programs SMART, SAINTPLUS, and SHELXTL. Enhanced versions of the proven and widely-accepted algorithms used by these programs (e.g., SAINT, SADABS, XPREP, XS, XM, XL, etc.) form the foundation of the APEX2 software.

2.1 Introduction to Client and Server Functions

The various programs within the Bruker Single-Crystal and Powder Diffraction Suite have a client-server relationship (Figure 2.1), in which the *server* program (BIS) executes commands given by one of several *client* programs (APEX2, APEX2Server, or BCP).

Figure 2.1 — Client and server relationships



2.1.1 Online and Offline Operation

The Suite can operate in *online* or *offline* mode.

In online mode, the Suite uses BIS to communicate with the instrument for performing experiments. To work in online mode, you must start BIS, your Suite program (i.e., APEX2). BIS will also start VIDEO when needed.

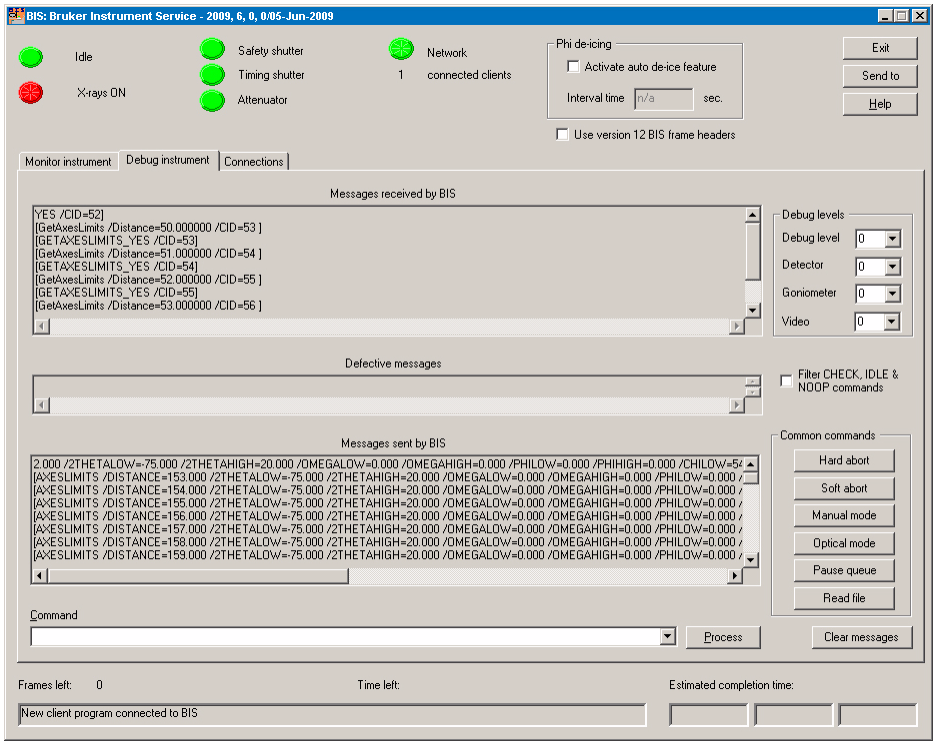
In offline mode, the Suite program is not connected to the instrument, but is still able to analyze and interpret existing data. To work in offline mode, you only need to start your Suite program.

2.2 Server Programs

2.2.1 The Bruker Instrument Service (BIS)

BIS acts as a server to the client programs, providing a link between the hardware and software. Once a connection is established, BIS executes hardware commands sent by the client programs. BIS can also be used as a service tool, displaying diagnostic messages during operation.

Figure 2.2 — BIS main window



2.3 Client Programs

2.3.1 APEX2 GUI and Modules

APEX2 is the main program that controls experiments and analyzes data. The user works with APEX2 though a variety of plug-ins, or *modules*, for different aspects of an experiment. The Suite includes a sample database that stores relevant data from each step in the experiment.

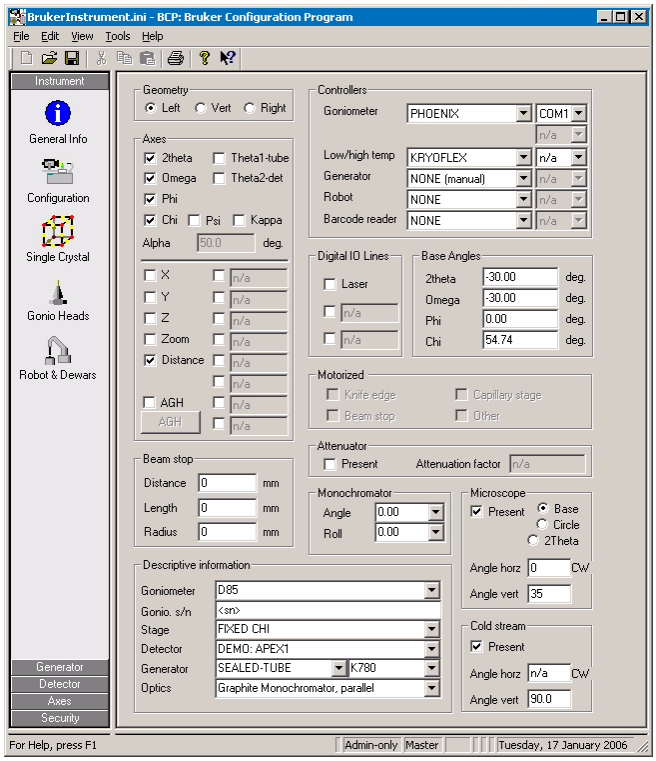
2.3.2 The Bruker Configuration Program (BCP)

BCP is a client program for BIS. It is used to configure BIS, as well as provide instrument control and alignment tools.

In BCP's main window (Figure 2.3), specific hardware parameters can be updated. BCP writes these values to the configuration file BrukerInstrument.ini, which is used by BIS. When changes are saved in BCP, BIS automatically reads in the new values.

See DOC-M86-EXX043 BCP and BIS Installation Notes, in addition to BCP's online help, for more information.

Figure 2.3 — BCP main window (Configuration screen)

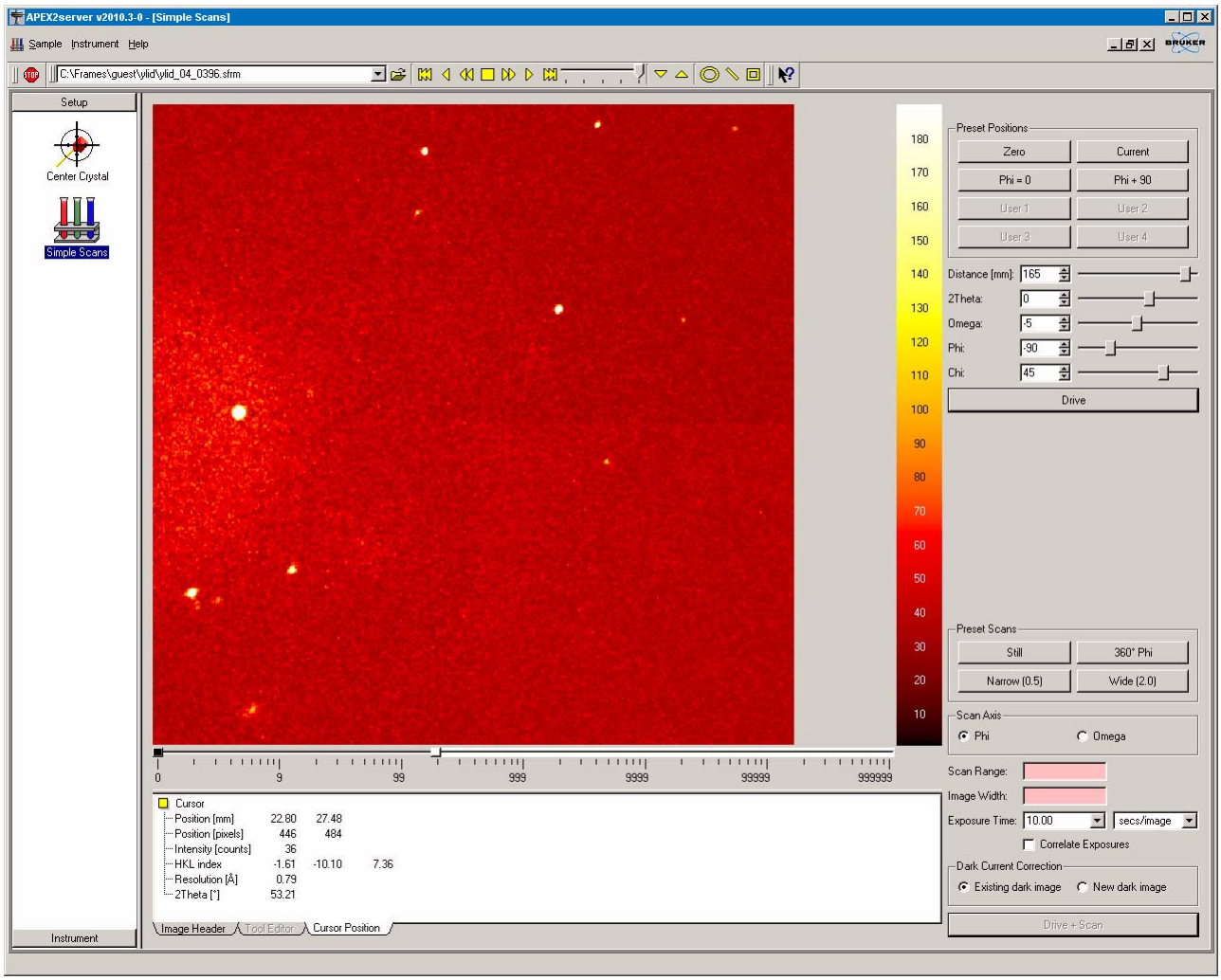


2.3.3 APEX2Server

APEX2Server is operated on the computer that controls the instrument. It provides functionality for quickly centering a crystal and determining its diffraction quality.

APEX2Server contains two main modules: “Center Crystal” and “Simple Scans” (see Figure 2.4).

Figure 2.4 — APEX2Server’s “Simple Scans” module



2.4 The Sample Database

The Sample Database is used for the storage of data generated by the Suite. It is designed to transparently handle data from all of the Suite's modules without intervention from the user.

The sample database is used internally by the Suite and is not available for user customization or manipulation. It is automatically started when the computer starts up, and it is automatically stopped before the computer shuts down.

2.5 License Considerations

You need a software license to activate your purchased software packages, options, and/or features.

The Suite requires a valid license file to operate. This file, "bn-license.dat", must be present in the root directory of drive C:\ for the software to start properly.

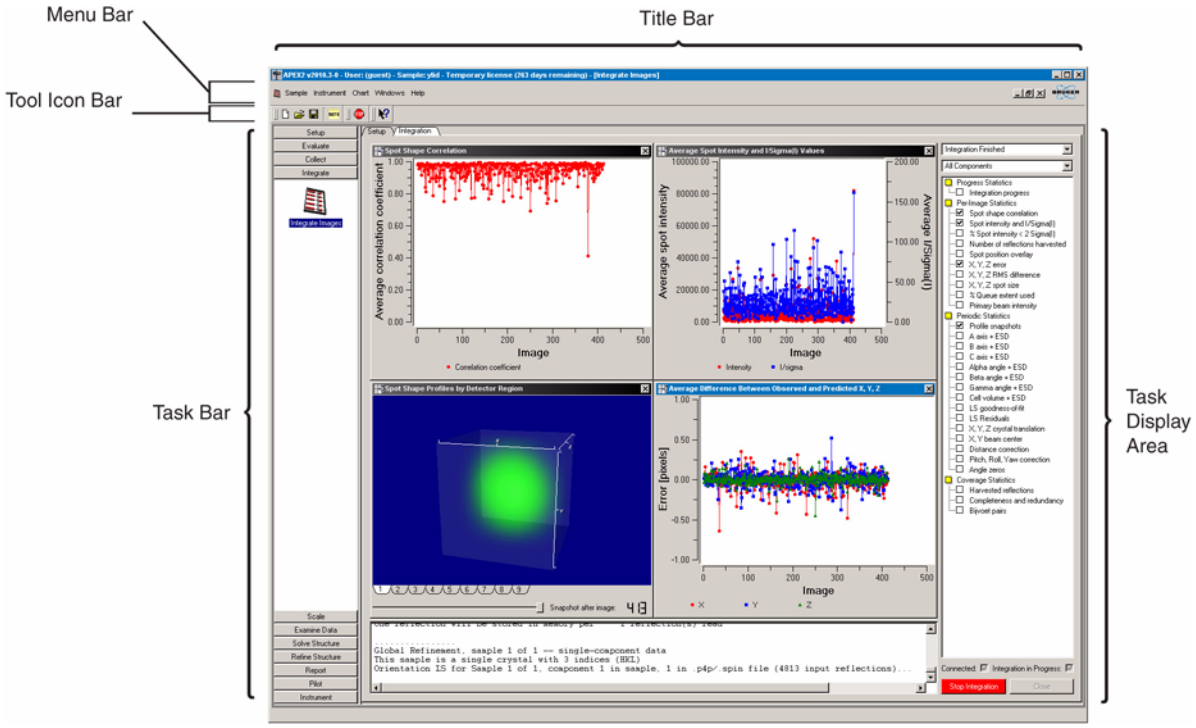
If the license file is not present or has expired, an error window will appear when you try to start your Suite program (i.e., APEX2). This window prompts you to run the License Manager application to obtain a valid license for the Suite.

For more information on licenses and License Manager, refer to DOC-M88-EXX099 License Manager User Manual.

2.6 The Main Window

The APEX2 graphical user interface (GUI) has one main window (Figure 2.5).

Figure 2.5 — Main window diagram



The main window is divided into five sections:

- Title Bar
- Menu Bar
- Tool Icon Bar
- Task Bar
- Task Display Area

NOTE: Throughout the interface, input fields with pink backgrounds indicate invalid entries. Disabled (i.e., grayed-out) fields indicate that a feature is not available (i.e., not supported or dependent upon the instrument configuration).

2.6.1 The Title Bar

The Title Bar displays the software name and version, user name, sample, license type, and active module. The right-hand edge of the Title Bar also contains the three Windows buttons for minimizing, restoring, and closing the main window.

2.6.2 The Menu Bar

The Menu Bar provides drop-down menus for a variety of file operations, image tools, and help files.

An icon for the currently-active module appears on the left-hand side of the Menu Bar. Clicking on this icon allows you to minimize, restore, resize, close, or move the currently-active window.

The Sample Menu

Figure 2.6 — Sample menu

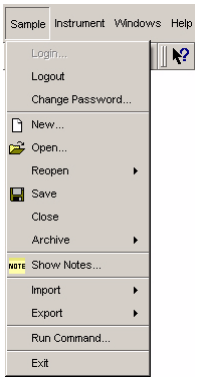


Table 2.1 — Sample menu commands

Sample menu command		Description
Login...		Opens a window for logging into the Sample Database using your username and password.
Logout		Logs out of the Sample Database.
Change Password...		Opens a dialog for changing your Sample Database password.
New...		Opens a dialog for creating a new sample in the Sample Database.
Open...		Opens a previously-saved sample.
Reopen		Reopens a recently-opened sample.
Save		Saves the current sample.
Close		Closes the current sample.
Archive	**Restore Archive...**	Restores an archived sample from a .zip file or CD.
	Archive Sample...	Archives the current sample to a .zip file or CD.
	Delete Samples...	Deletes samples from the Sample Database.
Show Notes...		Opens the “Sample Notes” window.
Import		Allows you to import crystal information contained in a .p4p or .spin file into the Sample Database.
Export		Allows you to export crystal data for the current sample as an .xml or .p4p file.
Run Command...		Opens a command prompt window.
Exit		Exits the application. This menu item has the same function as the button.

The Instrument Menu

Figure 2.7 — Instrument menu

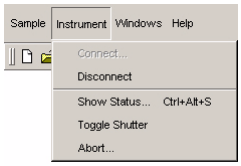


Table 2.2 — Instrument menu commands

Instrument menu command	Description
Connect...	Opens a connection to BIS.
Disconnect	If connected, disconnects from BIS.
Show Status...	Opens a window showing a summary of the current status of the instrument and the program's connection to it.
Toggle Shutter	Instructs BIS to send a shutter open or close command to the instrument. If all interlocks and safety circuits are satisfied, the shutter will open and close.
Abort...	Aborts all pending instrument operations.

The Windows Menu

Figure 2.8 — Windows menu

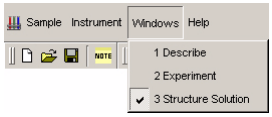


Table 2.3 — Windows menu command

Windows menu command	Description
Window selection	Displays a list of active windows. Any of these windows may be brought to the front by clicking its title.

The Help Menu

Figure 2.9 — Help menu

Table 2.4 — Help menu command

Help menu command	Description
About APEX2...	Displays APEX2's version and copyright information.

2.6.3 The Tool Icon Bar

Some icons on the Tool Icon Bar provide shortcuts to the options available through the Menu Bar.

Table 2.5 — Tool Icon Bar: menu bar shortcuts

Icon	Description
	Creates a new entry in the Sample Database.
	Opens an existing entry in the Sample Database.
	Saves the current information to the Sample Database.
	Shows the “Sample Notes” window
	Stops any instrument activity.
	“What’s this?” Context-sensitive help.

Other icons on the Tool Icon Bar are visible only when a frame is displayed.

Table 2.6 — Tool Icon Bar: displayed frame controls

Icon	Description
	Shows the currently-displayed frame’s filename. Click the drop-down triangle to choose previously-displayed frames.
	Opens a frame using a browse dialog.
	Displays the first frame in a run.
	Displays previous frame.
	Sequentially displays frames in reverse as a movie.
	If displaying frames as a movie, stops the movie.
	Sequentially displays frames as a movie.
	Displays the next frame.
	Displays the last frame in a run.
	Adjusts the rate of display when viewing frames as a movie.
	Displays frames from the next lower-numbered run.
	Displays frames from the next higher-numbered run.
	Draws a resolution circle.
	Draws a plotting line. The plot appears below the image.
	Selects a region of the frame.

2.6.4 The Task Bar

The Task Bar gives you access to all of the modules available in the Suite.

NOTE: If the Task Bar is not visible, there is no open sample. Choose **Sample > New...** or **Sample > Open...** to open or create a new sample.

Table 2.7 — “Setup” category

Icon	Name	Description
	Describe	Specify crystal size, color, shape, etc.
	Center Crystal	Manual and motorized crystal centering.
	Simple Scans	Provides tools for rapid crystal screening using phi and omega scans.

Table 2.8 — “Evaluate” category

Icon	Name	Description
	Determine Unit Cell	Determine unit cell and Bravais lattice type.
	Transform Unit Cell	Transform a unit cell.
	Reciprocal Lattice Viewer	3D visualization of the lattice projected in reciprocal space.
	View Images	View and analyze diffraction images.

Table 2.9 — “Collect” category

Icon	Name	Description
	Data collection strategy	Data collection strategy as in Collect.
	Data Collection Strategy	Simulate data collection and determine strategy.
	Experiment	Sequence editor for data collection experiments.
	Oriented Scans	Measure diffraction frames with the crystal aligned along axes.

Table 2.10 — “Integrate” category

Icon	Name	Description
	Integrate Images	Integrate diffraction data.

Table 2.11 — “Scale” category

Icon	Name	Description
	Crystal Faces	Determine Miller indices and distances of single-crystal faces.
	Scale	Scale intensities and perform absorption correction.

Table 2.12 — “Examine Data” category

Icon	Name	Description
	Space Groups and Statistics	Dataset analysis and manipulation.
	Compare Unit Cells	Compare unit cells to find twin laws.
	Precession Images	Create precession images based on measured frames.
	Diffraction Space Viewer	Create a 3D view in reciprocal space based on measured frames.
	Space Group Determination	Run XPREP.
	Find a Reflection	Find a reflection in measured frames.

Table 2.13 — “Solve Structure” category

Icon	Name	Description
	AUTOSTRUCTURE	Automatic solution and refinement with minimum user input.
	Structure Solution	Solve the structure.
	View Molecule	Visualize the molecule in 3D.

Table 2.14 — “Refine Structure” category

Icon	Name	Description
	Structure Refinement	Run XP, XL, or XSHELL.

Table 2.15 — “Report” category

Icon	Name	Description
	Generate Report	Use Bruker’s automatic report generation system.
	Generate Report	Run XCIF to generate a report.

Table 2.16 — “PILOT” category

Icon	Name	Description
	XRD² Eval	Display and integrate single and multiple frames.

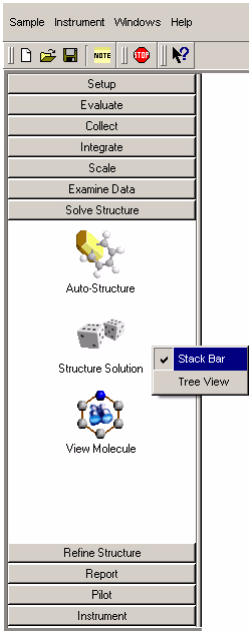
Table 2.17 — “Instrument” category

Icon	Name	Description
	System Calibration	Make calibration measurements.
	Unwarp and Convert Images	Convert frames between formats.
	Play Tic Tac Toe	Play a game of Tic Tac Toe!

Stack Bar and Tree View

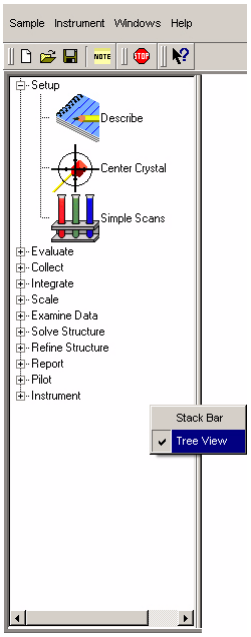
Right-click in the Task Bar to choose one of two views: Stack Bar or Tree View.

Figure 2.10 —Stack Bar view



In Stack Bar view, click one of the Task Bar categories to display only the icons belonging to that category. All other Task Bar icons will be hidden.

Figure 2.11 —Tree view



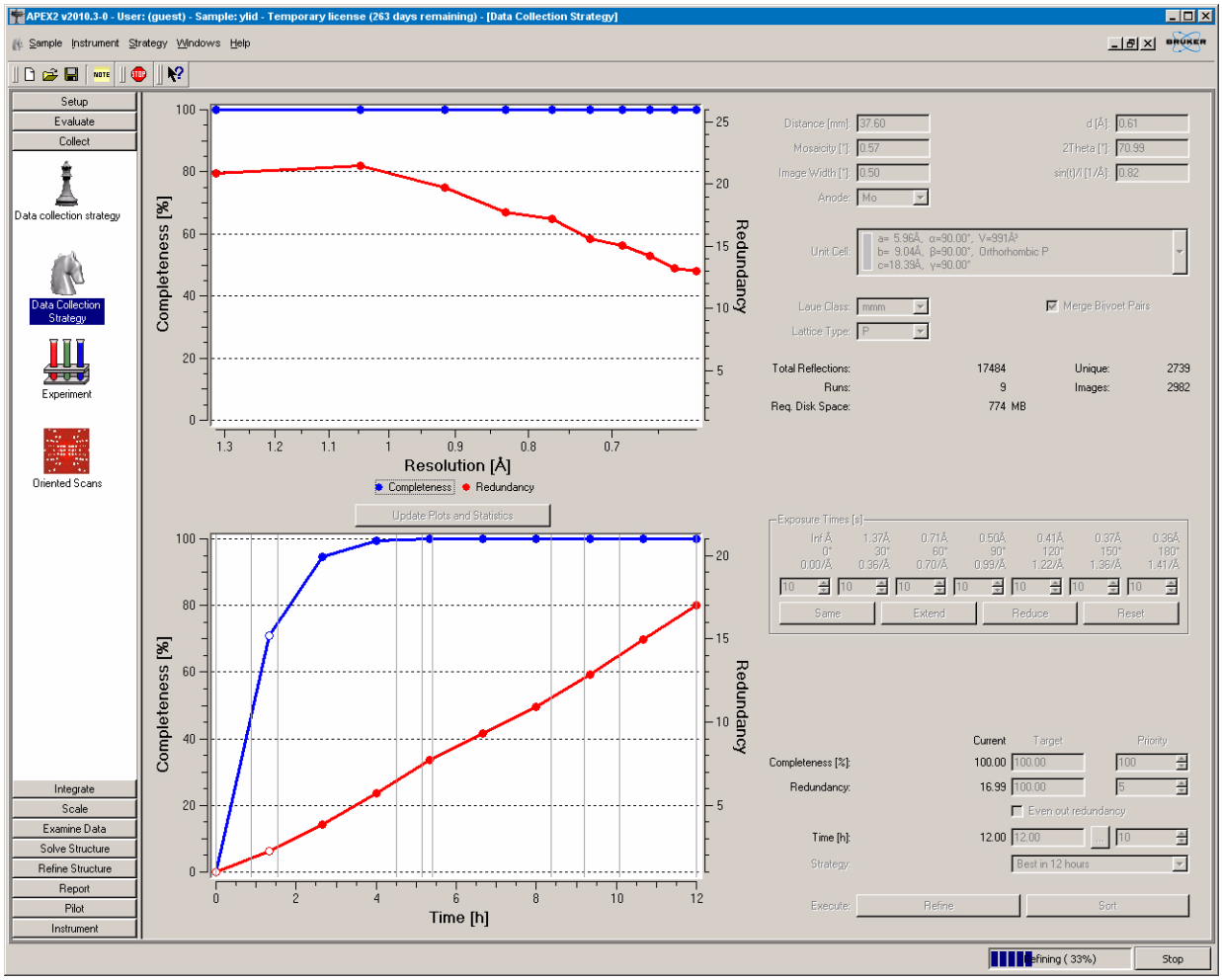
In Tree View, categories may be expanded to display their Task Bar icons by clicking the plus or minus sign next to the category name.

2.6.5 The Task Display Area

The Task Display Area is the main area for tasks, user input, and selected output. This area displays images and other output from the modules.

NOTE: In APEX2 Version 2, XSHELL refinement and XCIF report generation do not use the Task Display Area; they open in new windows. All other modules open in APEX2's Task Display Area.

Figure 2.12 —Task Display Area showing the Data Collection Strategy module



3 Startup and Shutdown

3.1 User Access

Bruker software allows different user accounts to have different levels of access to the instrument:

- Service
- Security
- Administrator
- User
- Non-user

You need “Bruker Instrument Administrator” rights to run BCP and “Bruker Instrument User” rights to run programs from the Suite. Typically, your system administrator will already have your user account configured or you will be using the default accounts as provided by Bruker AXS.

3.2 Startup

3.2.1 Start BIS

1. From the Windows Task Bar, choose **Start > Programs > Bruker AXS > Administration > BIS**.

   The BIS main window opens.

2. If you are asked to confirm any system parameters, do so and click **OK**.

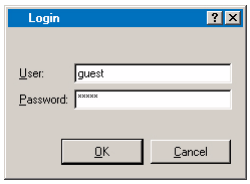
3.2.2 Start APEX2

1. From the Windows Task Bar, choose **Start > Programs > Bruker AXS > APEX2**.

3.2.3 Log In

1. At the “Login” dialog, enter your user name and password (as shipped by Bruker AXS, the default account is `guest` with password `guest`). Click **OK**.

Figure 3.1 — “Login” dialog



NOTE: If your setup does not require individual login accounts, you can configure the software to automatically log into a specified account.

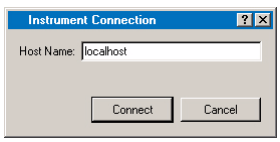
3.2.4 Connect to BIS

1. To use the software in online mode, choose **Instrument > Connect...**.

   The “Instrument Connection” dialog appears.

2. Enter the instrument host name (the dialog shows the default host). Click **Connect**.

Figure 3.2 — “Instrument Connection” dialog

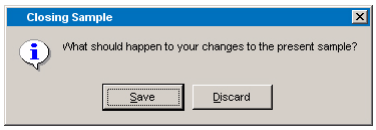


3.3 Shutdown

3.3.1 Shut Down APEX2

1. Log out of the Sample Database by choosing **Sample > Logout**.
2. In the “Closing Sample” window, choose whether to save or discard the changes made to the Sample Database.

Figure 3.3 — “Closing Sample” window



NOTE: The “Closing Sample” window appears even if no apparent changes were made to the Sample Database, because the date and time of the last access are held in the Sample Database and differ from the current date and time.

3. Choose **Sample > Exit**.

   APEX2 closes.

3.3.2 Shut Down BIS

NOTE: If BIS is running, it will prevent your computer from shutting down or rebooting.

1. Click the **Exit** button in the upper right-hand corner of BIS’ main window. You may be asked to place the generator into standby mode.
2. At the confirmation dialog, click **OK**.

   BIS closes.

This page intentionally left blank.

4 Crystal Centering and Screening

4.1 Centering the Crystal

To obtain accurate unit cell dimensions and collect high-quality data, position the crystal in the center of the X-ray beam and maintain this position for the entire experiment.

Your video microscope should be aligned so that the VIDEO software's reticle coincides with the goniometer center (for instructions on aligning the microscope to the center of the instrument, see DOC-M86-EXX024 Microscope Focus and Sample Alignment). If the reticle does not coincide with the goniometer center, you can still center the crystal. A successfully-centered crystal stays in the same place in the microscope's field of view in all orientations.

Also, the centering process can be greatly helped by lighting both the sample and background for maximum contrast.

NOTE: Use the thin end of the goniometer wrench to unlock the X, Y and Z locks at the beginning of the centering process and to lock them at the end. Locking needs only a very slight touch. The other end of the wrench is used to move the adjustment sleds. Do not overtighten the locks.

NOTE: Centering is often easier if the crystal is rotated to give a good view before the actual centering process is started (e.g., down an edge for a plate). To do this, drive to the initial centering position, loosen the crystal mounting screw, rotate the crystal to a suitable orientation, and then tighten the screw again.

4.1.1 Start the Center Crystal Module

1. Under the “Setup” category in the Task Bar, click the **Center Crystal** icon .

   The Center Crystal module opens.

4.1.2 Mount the Goniometer Head

1. Open the enclosure doors.
2. In the Center Crystal Module, click **Mount** to drive the goniometer to a convenient position for mounting the goniometer head.
3. Carefully remove the goniometer head containing the crystal from its case.

CAUTION

Use extreme care when handling the goniometer head to prevent damage to the crystal on the end of the small glass fiber.

4. Place the goniometer head onto its base on the phi drive. Line up the slot on the bottom of the goniometer head with the pin on the mounting base (Figure 4.1).

Figure 4.1 — Goniometer head bottom

5. Screw the goniometer head collar to the base so that the head does not move. Do not overtighten it (finger-tighten only).

Figure 4.2 — Huber goniometer head in detail

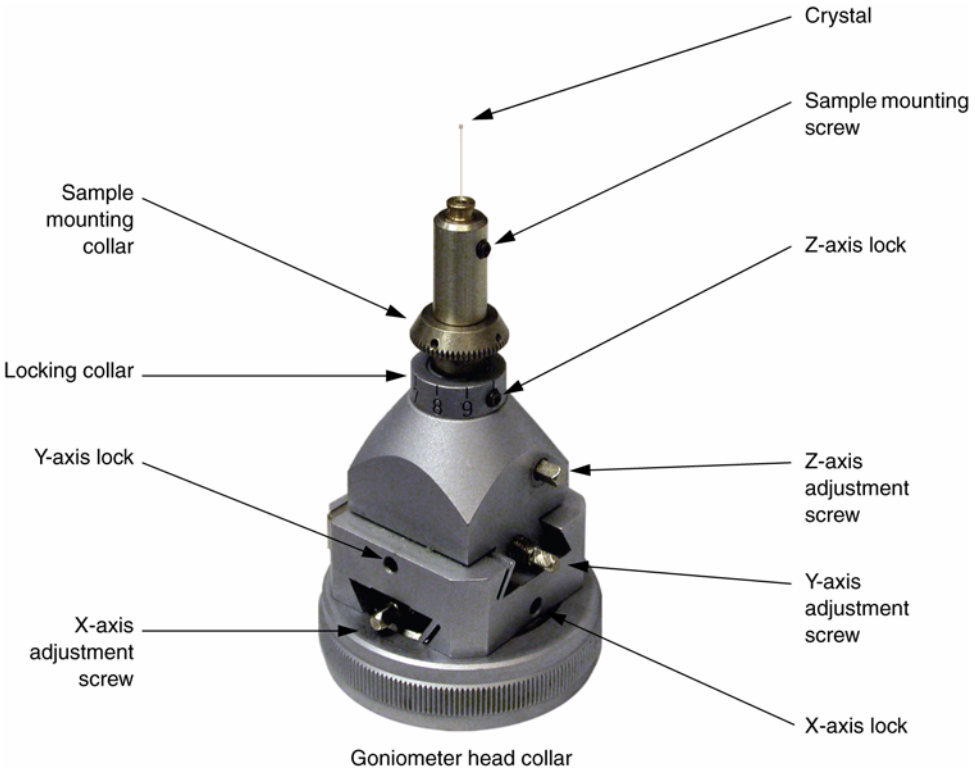
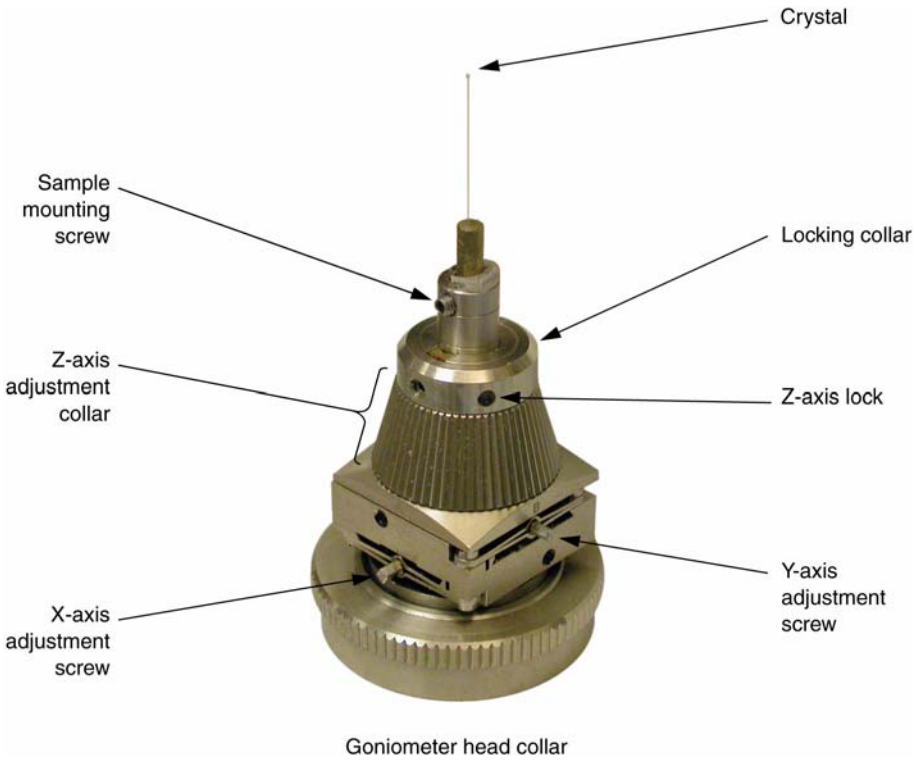


Figure 4.3 — Nonius goniometer head in detail



4.1.3 Center the Crystal on the KAPPA Series II Goniometer

1. Click the **Center** button.

   The goniometer head drives so that its Y and Z translation axes are positioned perpendicular to the microscope. The Y- and Z-axis adjustment screws should be facing the front of the diffractometer. If they are not, click **Spin Phi 180**.

Figure 4.4 — Center position, Y- and Z-axis adjustment screws

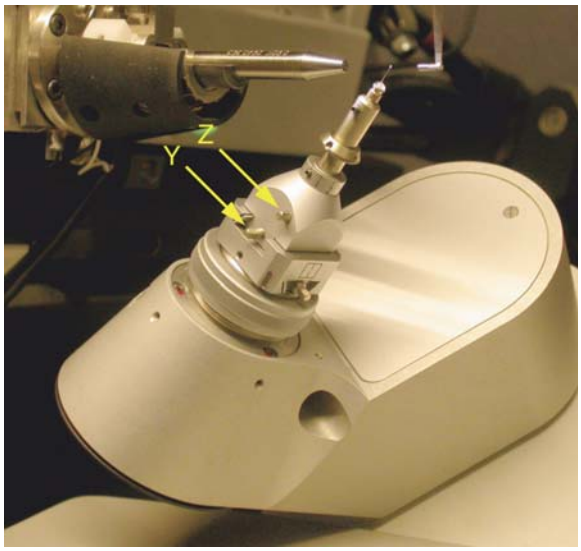
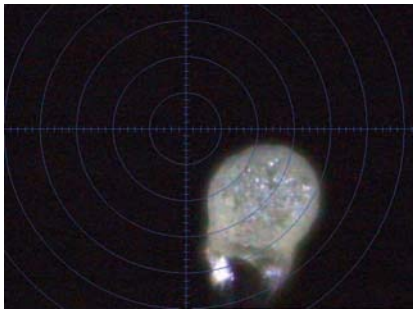


Figure 4.5 — Crystal initially mounted



2. Center the crystal in the video microscope reticle by making adjustments to the Y- and Z-axis adjustment screws.

Figure 4.6 — Centered crystal after Y- and Z-axis adjustments

3. Click **Spin Phi 180** and check that, after rotating 180°, the crystal appears in the same position in the microscope reticle. If the crystal does not appear in the same position:
    3.1. Use the adjustment screw facing you to remove half of the difference shown in the reticle (Figure 4.7 and Figure 4.8).
    3.2. Click **Spin Phi 180**.
    3.3. Repeat step 3.1 and step 3.2 until you are satisfied that the crystal remains in the same position in the microscope reticle when rotated by 180°.

NOTE: If the crystal consistently fails to appear in the same position when rotated by 180°, the position of the microscope reticle may need adjustment. For more information, refer to DOC-M86-EXX024 Microscope Focus and Sample Alignment.

Figure 4.7 — Error in Y-axis

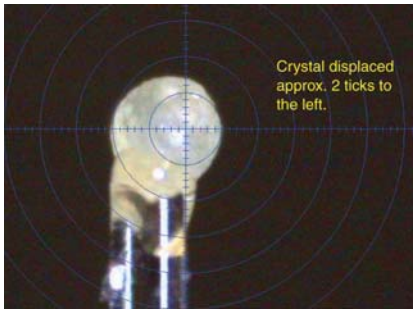
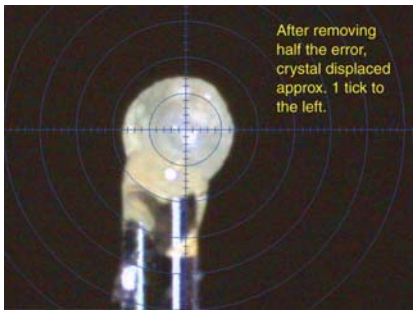


Figure 4.8 — Example: error removed by half



4. Click **Spin Phi 90**.

   Phi rotates so that the X-axis adjustment is facing forward. Any error in the crystal's position along the X-axis will now appear in the microscope.

Figure 4.9 — Center position, X-axis adjustment screw

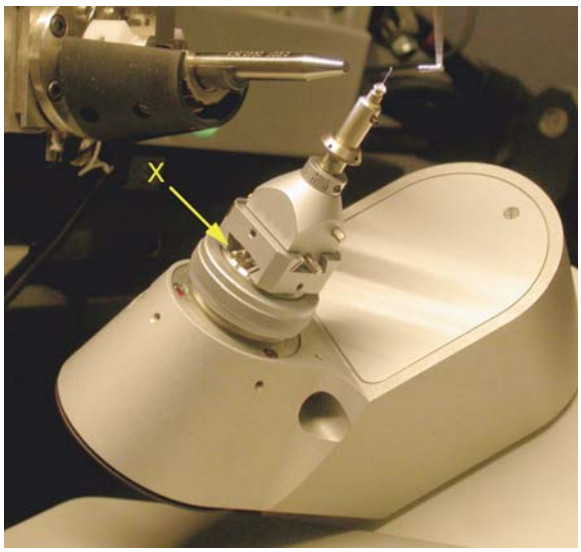


Figure 4.10 —X-axis error after spinning phi 90°

5. Center the crystal in the video microscope reticle by making adjustments to the X-axis adjustment screw.

Figure 4.11 —Centered crystal after X-axis adjustments

6. Alternately click **Spin Phi 180** and **Spin Phi 90** to verify that the crystal stays in the same place in the microscope reticle through all motions of phi. If the crystal fails to stay in the same position as phi is rotated, make adjustments to the axes by repeatedly removing half the error as in step 3.
7. Click the **Left** button. The goniometer drives to place the fiber horizontal and to the left. Note the height of the crystal in the video microscope reticle.

Figure 4.12 —Left position

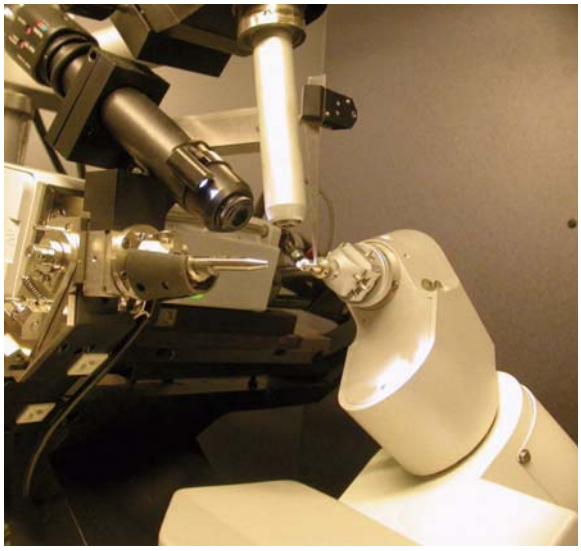
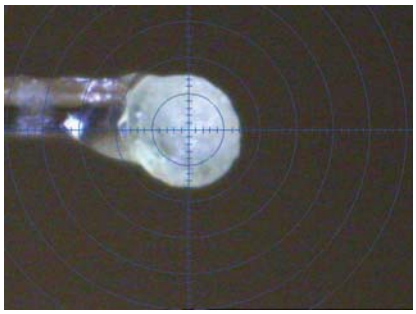


Figure 4.13 —Crystal in left position



8. Click the **Right** button.

   The goniometer drives to place the fiber horizontal and to the right.

Figure 4.14 —Right position

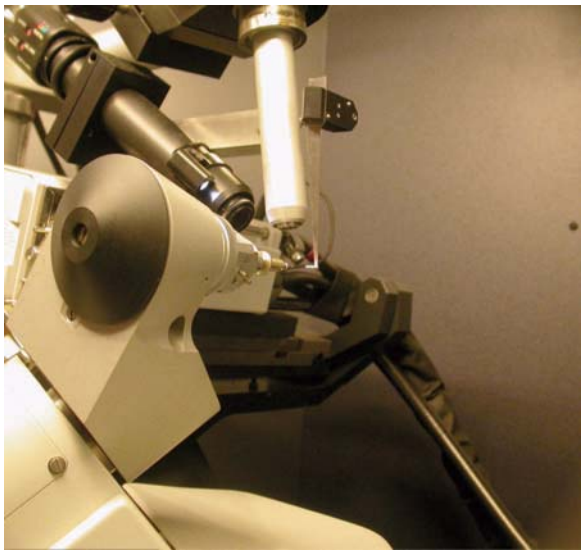
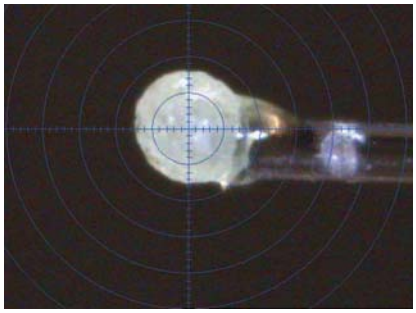


Figure 4.15 —Crystal in right position



9. Check that the crystal height is the same as the height you noted in step 7.

   9.1. If the height is the same, proceed to step 10.

   9.2. If the height is not in the same place, adjust to remove half of the difference, click **Spin Phi 180**, and repeat step 7 through step 9.

10. Click the **Top** button.

    The goniometer drives so that the phi axis is pointing directly into the microscope.

Figure 4.16 —Top position

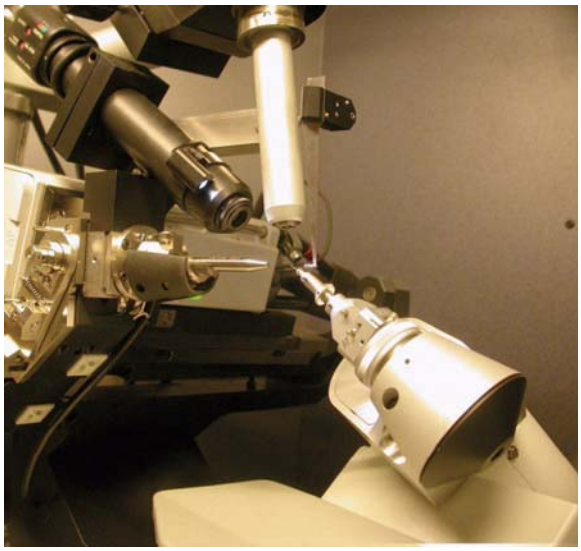
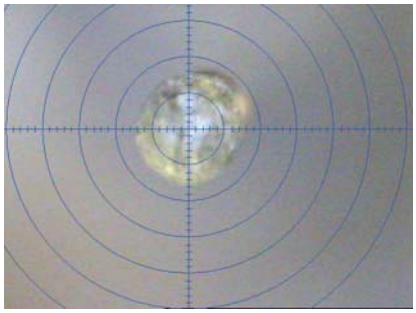


Figure 4.17 —Crystal in top position



11. Click **Spin Phi 180** a few times to verify that the crystal stays in the same position as Phi rotates. If the crystal does not remain centered, go back to step 1.
12. Click **Center** to drive the goniometer back to the center position.

The crystal is now centered on the KAPPA Series II goniometer.

4.1.4 Center the Crystal on the D85 Goniometer

1. Click the **Right** button.

   The crystal and goniometer head drive to a position perpendicular to the microscope. To center the crystal, make adjustments to the height with the Z-axis adjustment.

Figure 4.18 —Right position, Z-axis adjustment screw

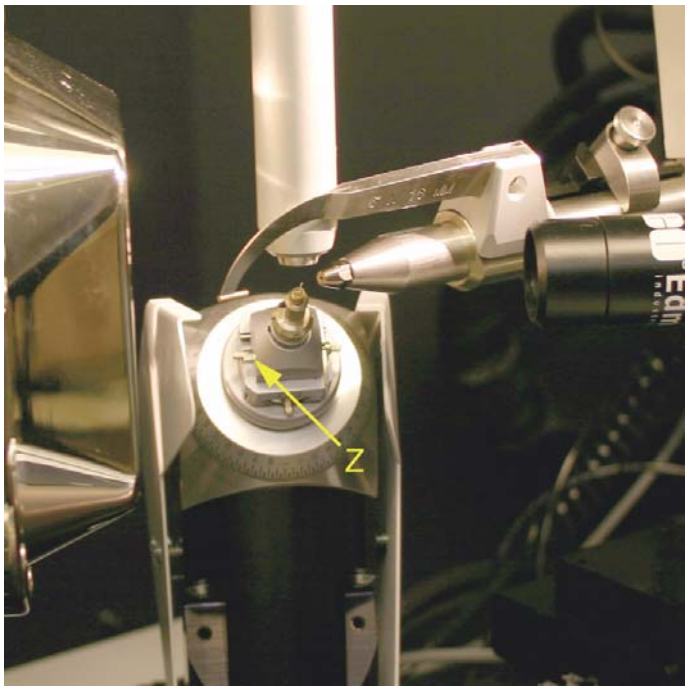
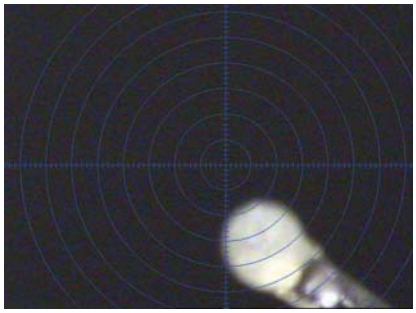


Figure 4.19 —Initial mounted crystal



2. Click the **Center** button.

3. Move the crystal so that it is centered in the microscope reticle by adjusting the X- or Y-axis translation adjustment screw that is perpendicular to the microscope axis and facing you.

Figure 4.20 —Center position, Y and Z adjustments

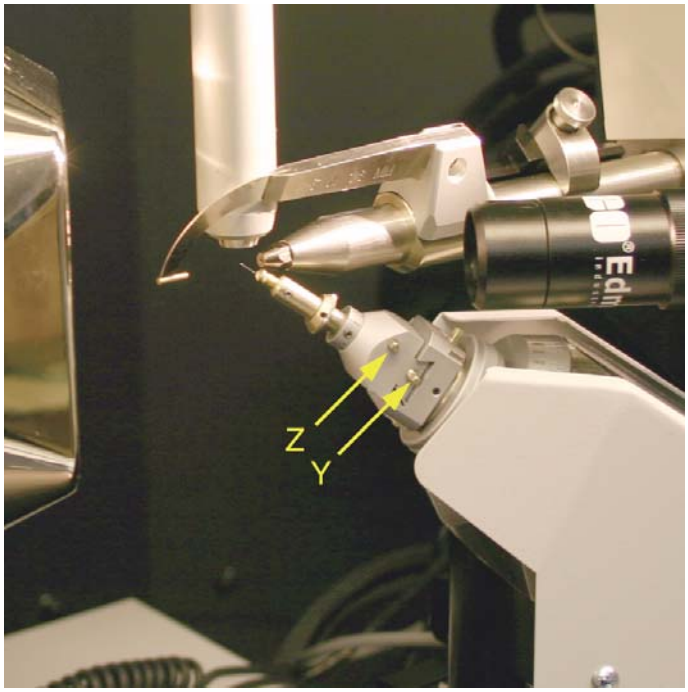


Figure 4.21 —Initial center position before X- or Y-axis adjustment

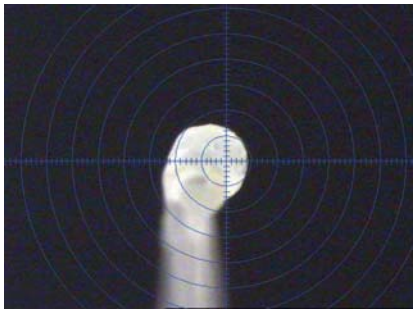
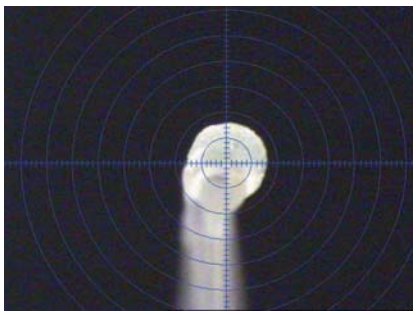


Figure 4.22 —Crystal after X- or Y- adjustments



4. Click **Spin Phi 90**. Remove half of the difference with the adjustment screw that is facing you.

Figure 4.23 —Example: error in X-axis

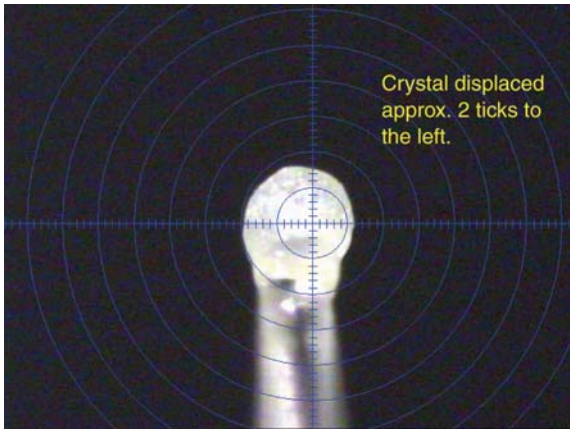
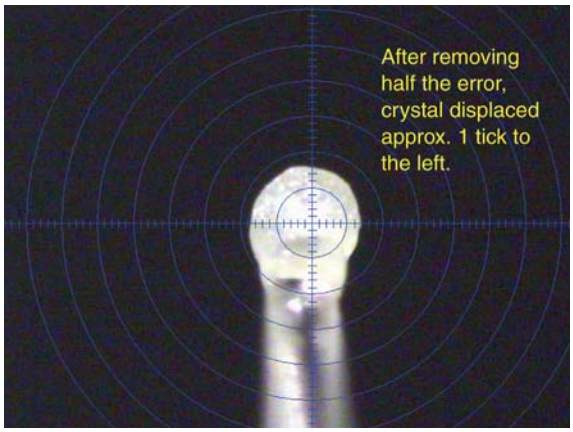


Figure 4.24 —Example: error removed by half



5. Click **Spin Phi 180**. Remove half of the difference with the adjustment screw that is facing you.
6. Click **Spin Phi 180** again.
7. If the crystal is centered, click **Spin Phi 90**.
8. If the crystal is not centered, adjust to remove half of the difference and click **Spin Phi 180**. Repeat until the crystal is centered.

9. Click **Spin Phi 90**. If centered, adjust the height. If not centered, repeat step 4 through step 8 until it is centered.

Figure 4.25 —Height adjusted

10. Click the **Left** button. Adjust to remove half of the difference. Adjust the height.

Figure 4.26 —Left position

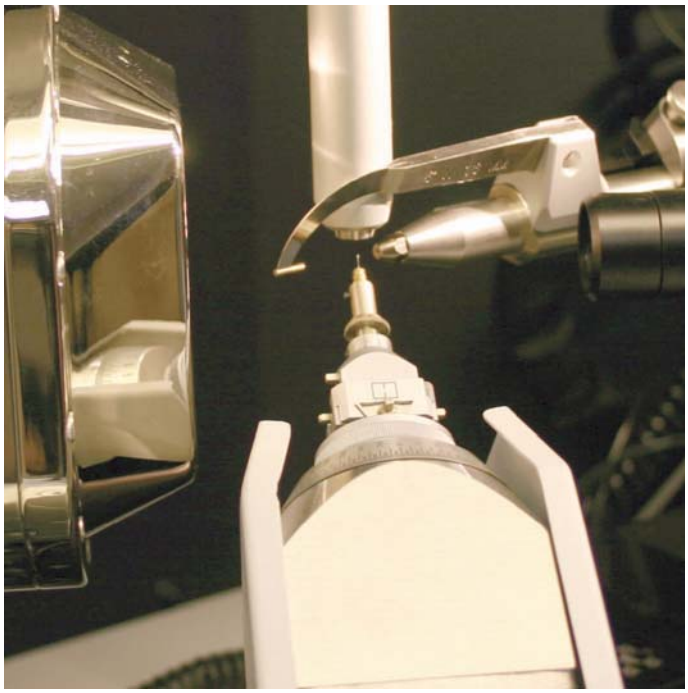
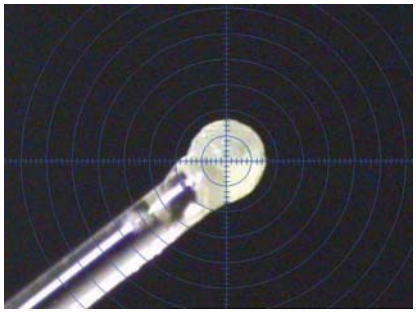


Figure 4.27 —Check height in left position



11. Click the **Right** button. Adjust the height. Adjust to remove half of the difference.

Figure 4.28 —Right position

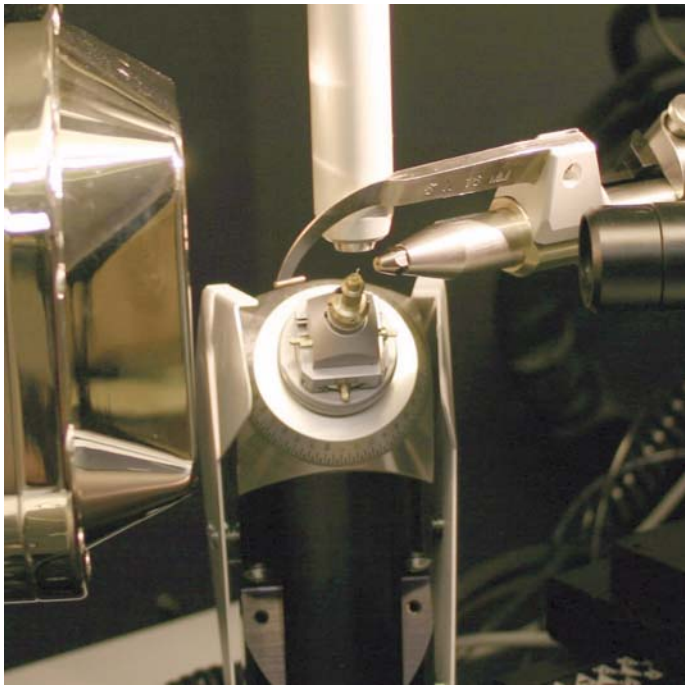
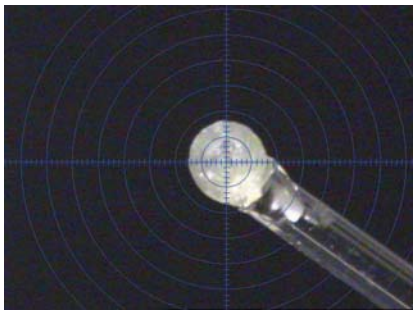


Figure 4.29 —Check height in right position



12. If a height adjustment was made in step 7 or step 8, repeat those steps to check the height. If the height is correct, repeat step 3 through step 6 to check the centering. If no height adjustment was made, the crystal is centered.

The crystal is now centered on the D85 goniometer.

4.2 Checking Crystal Quality

A number of tools provide information about the current image. These tools, present throughout the Suite, can help determine the diffraction characteristics of a crystal.

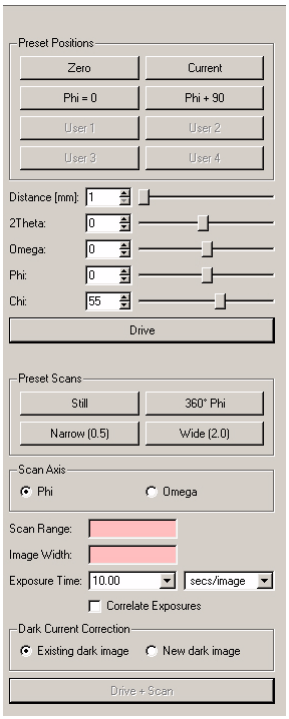
4.2.1 Start the Simple Scans Module

1. In the Task Bar's "Setup" category, click the **Simple Scans** icon .

   The Simple Scans module opens.

4.2.2 Set up the Scan

Figure 4.30 —Simple Scans menu



1. Set the goniometer to the desired position either by using the preset position buttons, adjusting the sliders, or manually editing the goniometer angle values.

NOTE: The **User** positions may be defined in the bn-config.py file.

2. Click **Drive** to initiate the requested movement.

NOTE: You must click the **Drive** button to move the goniometer to the new position. If the button is gray, the requested position violates the software limits.

3. Set up the scan parameters either by using the preset scan buttons or manually editing the values.
4. Choose whether to use the existing dark current correction or a new one by clicking the appropriate radio button.
5. Click **Drive + Scan** to start the scan.

Collect a 360-degree Phi Scan

1. Click the **360° Phi** button.

   A value of `360.00` is displayed in both the “Scan Range:” and “Scan Width:” fields.
2. If necessary, enter an exposure time (the default exposure time is usually sufficient).
3. To start the scan, click the **Drive+Scan** button.

Collect a Still Scan

1. Click the **Still** button.

   A value of `0.00` is displayed in both the “Scan Range:” and “Scan Width:” fields.
2. Enter an exposure time.

   You can select the units to be either seconds/frame or seconds/degree.
3. To start the scan, click the **Drive+Scan** button.

   An example of starting goniometer positions for sample screening is shown in Table 4.1. If the reflections are overlapping, increase the DX distance. If there are reflections that extend out to the edge of the image, increase the 2-theta angle to determine how well the crystals diffract (10° intervals work well).

NOTE: If the low-temperature device’s nozzle is perpendicular to the goniometer base and chi is near zero for a long period of time, ice may form on the goniometer head. Whenever BIS becomes idle, it will automatically drive the crystal to chi = 45° to eliminate icing.

Table 4.1 — Starting goniometer positions

Goniometer	DX (mm)	2theta (°)	Omega (°)	Phi (°)	Chi (°)
D85	60	0	0	0	54.74 (fixed)
KAPPA Series II	60	0	0	0	45

Collect a Narrow or Wide Rotation Scan

1. Choose the desired rotation axis by clicking either the **Phi** or **Omega** radio button.
2. Choose either **Narrow** or **Wide** for the rotation angle.

   The same values will appear in the editable “Scan Range:” and “Image Width:” fields.

   For example, if you want to collect 3° of data at 0.5° intervals, choose **Narrow** and change `0.5` in the “Scan Range” field to `3.0`. The “Image Width” field can also be adjusted in the same way.
3. Adjust the exposure time as you would for a still image.
4. Click the **Drive + Scan** button.

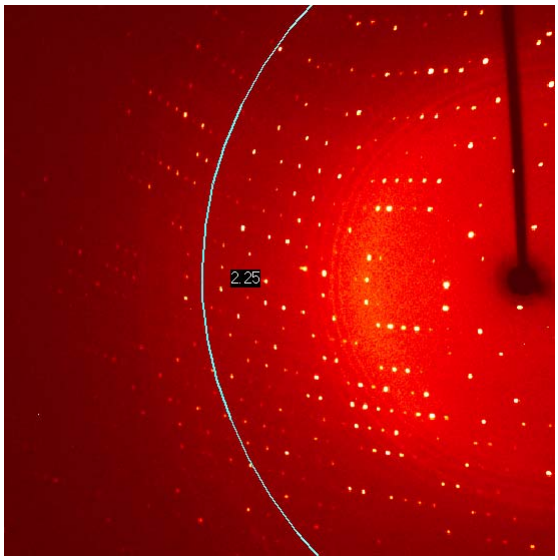
   If the **Correlate Exposures** checkbox is activated, the image will be taken in two parts and added together. If the **New dark image** radio button is activated, the system will generate a new dark image with a duration equal to the current exposure time.

4.2.3 Use the Resolution Circle Tool

1. Click the Resolution Circle tool in the Tool Icon Bar to activate it.
2. Click and drag the mouse to draw the resolution circle.

   The circle will extend outward from the beam center and display the resolution at the circle edge (the ring is always drawn from the direct beam position).

Figure 4.31 —Drawing a resolution circle



3. To hold the position, release the button.
4. To deactivate the Resolution Circle tool, click its icon in the Tool Icon Bar.

4.2.4 Use the Line Tool

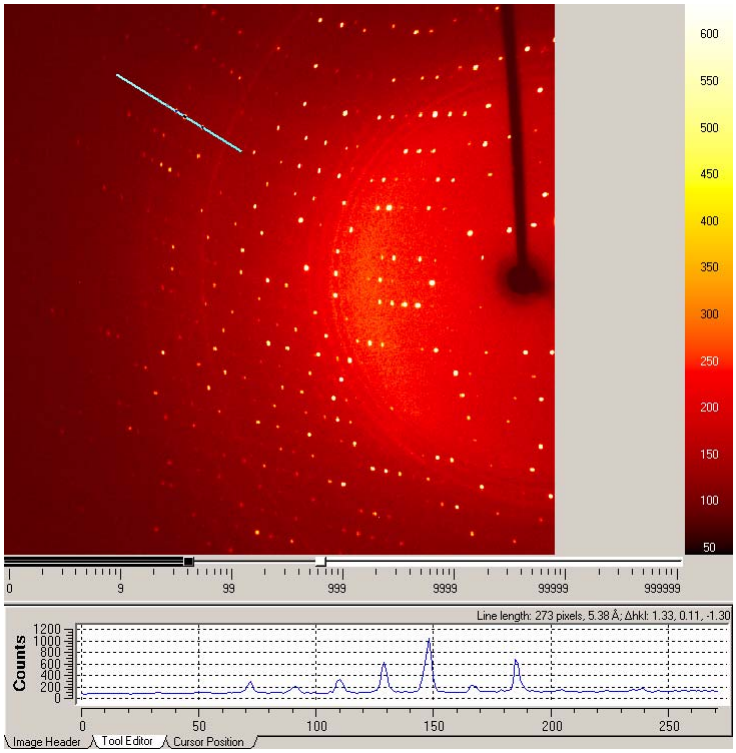
A quick way to check whether reflections are overlapping is to use the Line Tool.

A chart showing the counts for each peak under the line is displayed at the bottom. The length of the line in pixels and angstroms is also shown.

1. Click the Line tool in the Tool Icon Bar to activate it.
2. To draw the line, click and drag the mouse.

   When you release the button, the length of the line remains fixed and it can be placed anywhere on the image.

Figure 4.32 —Drawing a line



3. To deactivate the Line tool, click its icon in the Tool Icon Bar.

4.2.5 Plot a Rocking Curve

The Box tool allows the rocking curve determination of a spot. The full-width half-maximum for a peak's rocking curve is a good estimate of the peak's mosaic spread. Looking at a few peaks away from the Lorentz region gives a rough idea of the overall mosaicity.

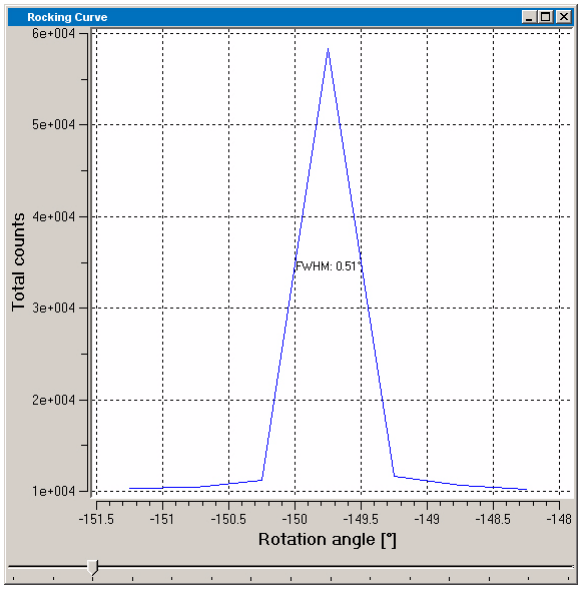
1. Click the Box tool in the Tool Icon Bar to activate it.
2. Click and drag to draw the box.

   If necessary, zoom into the selected area by right-clicking and choosing **Zoom+**.
3. Draw a box around a single reflection, right-click, and choose **Rocking Curve**.

   A graph appears displaying the total counts as a function of the rotation angle.

   You typically need around 1.5° of data on either side of a scan to sample several peaks. If the peak begins at and returns to the background level, the full-width half-maximum value will be displayed. If additional scans are available, the slider bar at the bottom of the graph allows the angular range to be increased.

Figure 4.33 —Rocking curve



4. To close the "Rocking Curve" window, click the button.
5. To deactivate the Box tool, click its icon in the Tool Icon Bar.

4.2.6 View Peak Profiles in 3D

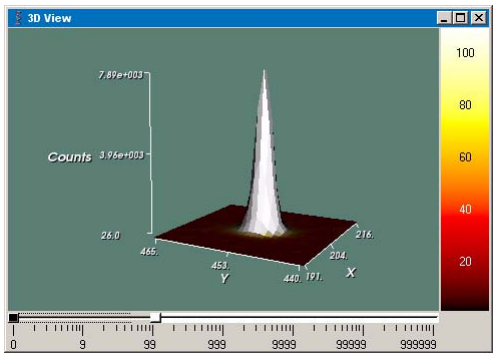
1. Click the Box tool in the Tool Icon Bar to activate it.
2. To draw the box, hold the left mouse button down and drag the mouse.

   If necessary, zoom into the selected area by right-clicking and choosing **Zoom+**.
3. Draw a box around a peak or group of peaks, right-click, and choose **3D-View**.

   A 3D profile of the box region appears.

   The profile can be rotated by holding down the left mouse button and dragging the mouse. the display colors can be changed by right-clicking the contrast scale.

Figure 4.34 —3D View



4. To close the “3D View” window, click the button.
5. To deactivate the Box tool, click its icon in the Tool Icon Bar.

4.2.7 Examples of Poor-Quality Crystals

Figure 4.35 shows a 360° phi scan on what is likely a poor-quality crystal. The spot shape is poor and the spots tend to run together. The obvious bands on the image suggest that the crystal is nearly aligned along an axis; however, spots will always apparently run together in a nearly-aligned crystal mounting. Figure 4.36 is much more convincing proof of a poor-quality crystal.

Figure 4.35 —Possible poor-quality crystal, 360° phi scan

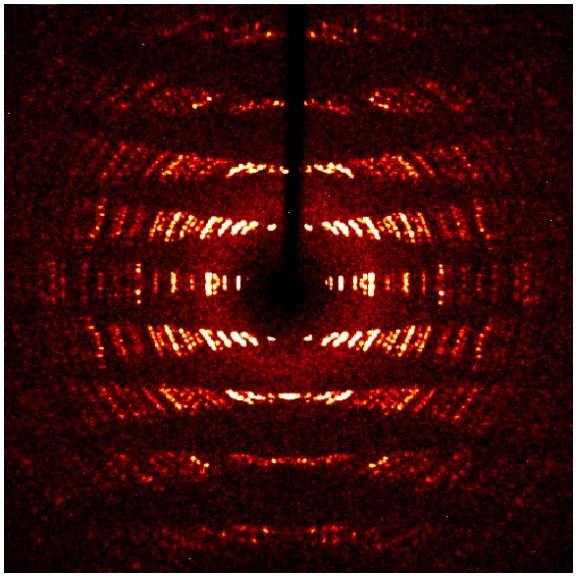


Figure 4.36 shows a 2° phi scan on a poor-quality crystal. The spot shape is poor, and some spots seem split.

Figure 4.36 —Poor-quality crystal, 2° phi scan

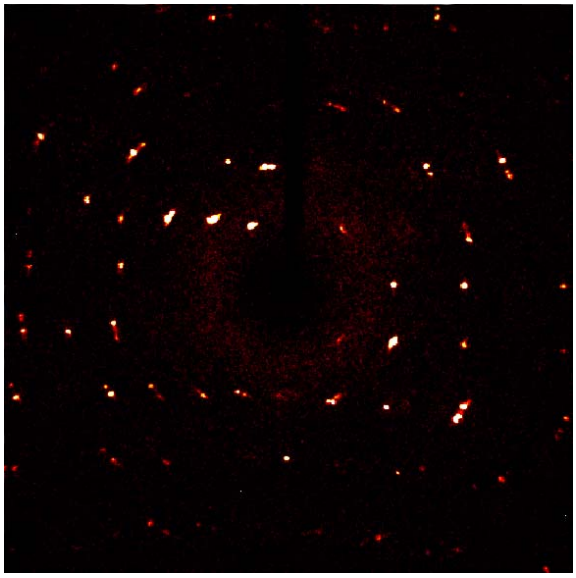


Figure 4.37 shows a 2° phi scan on a poor-quality crystal at +90 in phi. The spot shape is poor, and the spots are very close together.

Figure 4.37 —Poor-quality crystal, 2° phi scan, phi=+90°

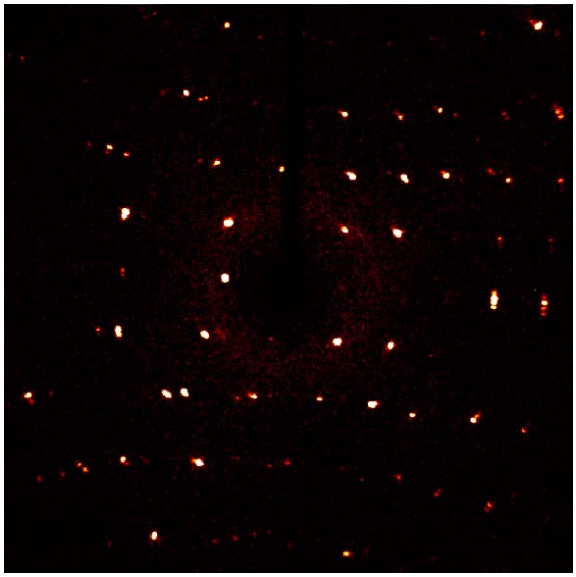
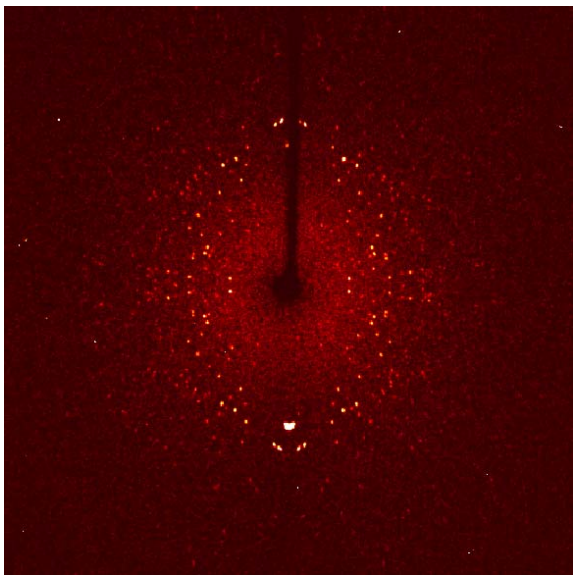


Figure 4.38 shows a 360° phi scan on a small crystal. The diffraction power of the crystal is poor, but with longer exposure times this is a reasonable candidate for data collection.

Figure 4.38 —Good-quality crystal but weak diffractor



5 Data Collection

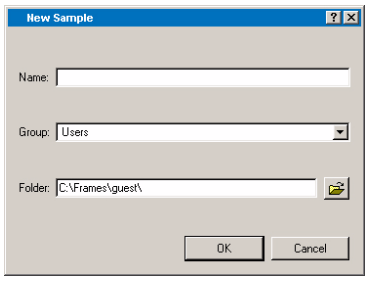
In the data collection example that follows, we will use an ylid crystal with the sample name `ylid` (entered in Section 5.1).

5.1 Create a New Sample

1. Choose **Sample > New...**.

   The “New Sample” dialog opens.

Figure 5.1 — “New Sample” dialog



2. In the “New Sample” dialog, enter the sample name and choose an access group for your sample (if desired). The Suite will automatically create a directory for data storage.
3. Click **OK**.

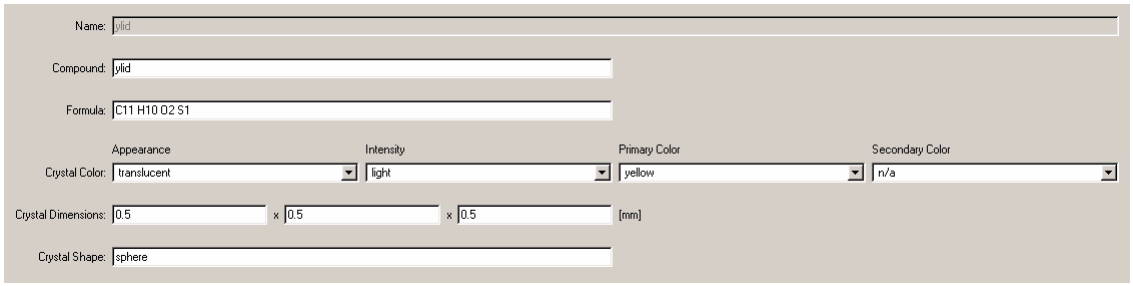
   The Task Bar appears with the “Setup” category open.

5.2 Describe the Sample with the Describe Module

1. In the Task Bar’s “Setup” category, click the **Describe** icon .

   The Describe module opens.
2. Fill in the fields.

Figure 5.2 — Describe module (filled in)



3. Close the Describe module by clicking the button on the right-hand side of the Menu Bar.

   The data is automatically saved to the Sample Database.

5.3 Determining the Unit Cell

Unit cell determination is performed in several steps:

1. Data collection
2. Harvesting of reflections from collected frames
3. Indexing of harvested reflections
4. Bravais lattice type determination
5. Refinement

These steps are integrated in the Determine Unit Cell module, which also contains a one-click solution for fully-automated unit cell determination.

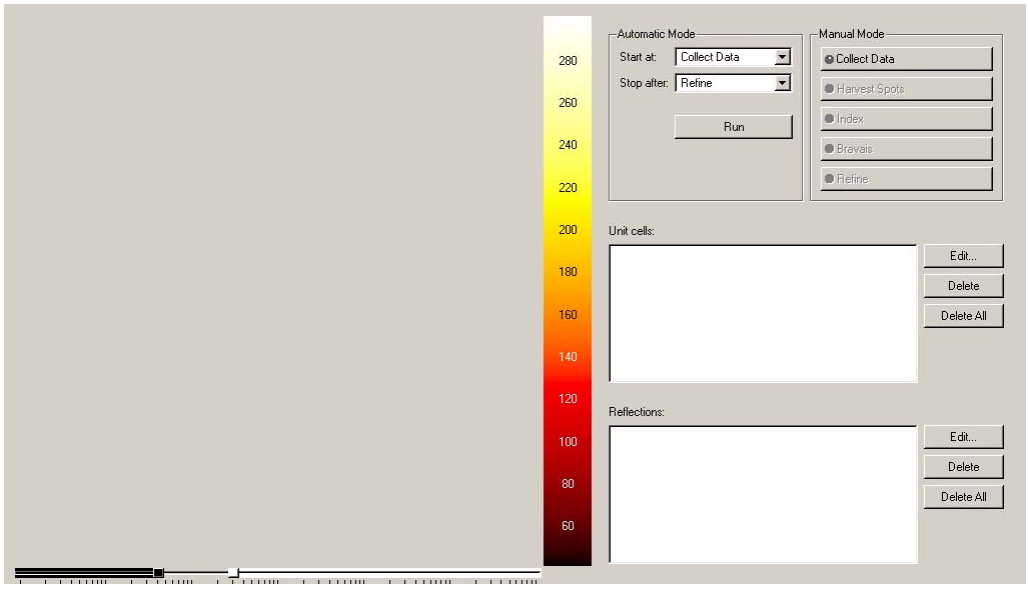
The upper right-hand corner of the Determine Unit Cell module contains two sections:

- Automatic Mode - for fully-automated unit cell determination (Section 5.3.2); and
- Manual Mode - for unit cell determination with user-defined parameters (Section 5.3.3).

NOTE: The Determine Unit Cell module automatically determines crystal mosaicity and provides a table of exposure time versus diffraction limit (Figure 5.3, lower right-hand corner).

NOTE: This table is strictly informational and its contents are not used when determining exposure times for a data collection strategy. However, you can use the information in the table to manually set exposure times for your maximum desired resolution.

Figure 5.3 — Determine Unit Cell module (initial view)



5.3.1 Start the Determine Unit Cell Module

1. In the Task Bar's "Setup" category, click the **Determine Unit Cell** icon .

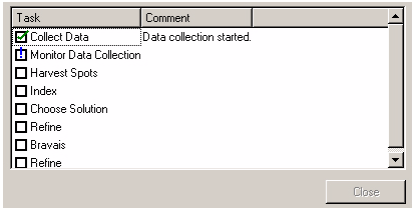
   The Determine Unit Cell module opens.

5.3.2 Determine the Unit Cell in Automatic Mode

1. From the **Start At:** pull-down menu, select the step at which you want to begin automatic unit cell determination (useful if you want to find a unit cell from frames that have already been collected).
2. From the **Stop After:** pull-down menu, select the step at which you want to stop automatic unit cell determination.
3. Click **Run**.

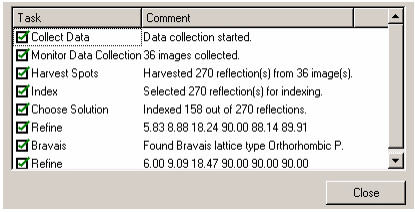
   Automatic data collection begins, and collected frames appear in the Information Display Area. The area containing the Automatic and Manual mode options changes to a list showing the system's progress through the steps of unit cell determination.

Figure 5.4 — Automatic Mode progress list



When automatic unit cell determination is complete, the progress list will show a check mark and comments for each completed step (Figure 5.5).

Figure 5.5 — Automatic Mode progress list (complete)



4. Click the **Close** button to close the progress list and return to the main Determine Unit Cell window.

   The window's right-hand side displays the unit cell parameters, along with options to edit or delete the unit cell and reflection list if you wish to perform additional operations in Manual Mode.

NOTE: If desired, you can set up your own runs for the Determine Unit Cell module by using BCP's **Single Crystal** function (in the "Instrument" category) or by editing the bn-config.py configuration file.

5.3.3 Manual Mode

The Determine Unit Cell Module's Manual Mode functions allow you a great degree of control over unit cell determination. Manual Mode consists of five sections, each with its own menu, corresponding to the five stages of unit cell determination:

1. Collect Data
2. Harvest Spots
3. Index
4. Bravais
5. Refine

Collect Data

Figure 5.6 — Collect data menu

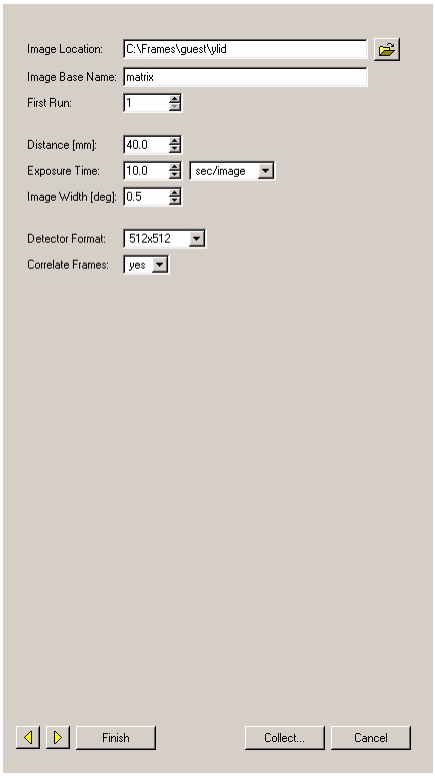


Table 5.1 — Collect Data menu items

Menu Item	Function
Image Location:	Location where collected frames will be saved.
Image Base Name:	Text string that is appended to each frame's filename and is used to identify frames by their filenames.
First Run:	Choose which run number will be the first run; useful for adding runs without overwriting previous ones.
Distance [mm]:	Detector distance.
Exposure Time:	Duration, in seconds, of each frame.
Image width [deg]:	Distance, in degrees, that the scan axis travels over the course of a single exposure.

Menu Item	Function
Detector Format:	Resolution of the frames collected by the detector.
Correlate Frames:	Two frames are taken (each with half the duration of the overall Exposure Time), to correct for spurious events appearing on individual frames.
[left arrow]	Go to the previous step in the unit cell determination process.
[right arrow]	Go to the next step in the unit cell determination process.
Finish	Finish the sequence of steps in the unit cell determination process, beginning with the current step. Perform any tasks that are necessary.
Collect...	Proceed with data collection according to the options set in the preceding menu items.
Cancel	Leave Manual Mode and return to the initial Unit Cell Determination page, performing no operations and ignoring any changes made to menu items.

Harvest Spots

Figure 5.7 — Harvest Spots menu

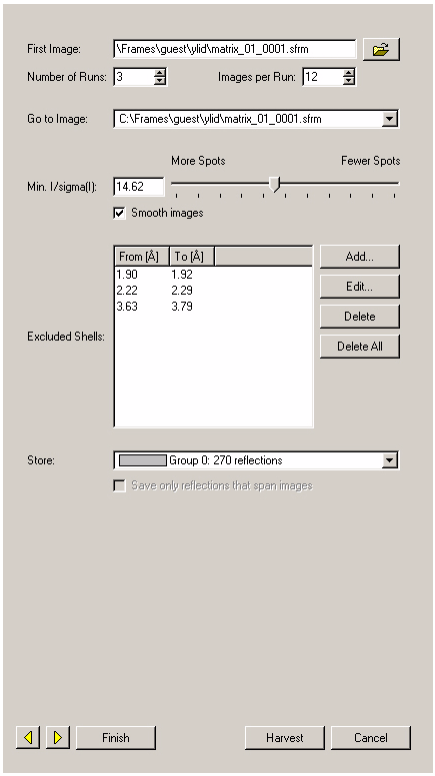


Table 5.2 — Harvest Spots menu items

Menu Item	Function
First Image:	Select the first image in the group of images to be examined for spots.
Number of Runs:	Number of runs to be examined.
Images Per Run:	Number of images to be examined in each run.
Go to Image:	Select a frame filename from this drop-down menu to display the frame in the Image Information Area.
Min. I/sigma(I):	Adjust the criterion for harvesting a spot based on its pixels' intensity versus their standard deviation. Slide the slider between "More Spots" and "Fewer Spots" to vary the minimum I/sigma(I).
Smooth images	A Gaussian filter is applied to the frames prior to harvesting, which reduces the noise and eliminates falsely harvested pixels. For very weak data, however, the Smooth images function can interfere with successful harvesting.
Excluded shells:	Add, edit, or delete resolution shells to be excluded from the harvesting process, for example in the case of rings caused by ice or amorphous diffractors (the three inner resolution shells for water ice appear as defaults). Excluded shells are defined by their starting and ending resolution (which may be found by placing the mouse cursor on a displayed frame and referring to the Resolution [Å] field in the Cursor Position Tab).

Menu Item	Function
Store (Reflection Group Combo Box):	The Reflection Group Combo Box shows a list of reflection groups to choose from. For each group, the group name and number of reflections in the group are displayed. If, instead of the number of reflections, an entry is labeled empty, this indicates an unassigned entry. Choosing the empty entry creates a new group instead of appending to the current one. A color is associated with each reflection group. The color is displayed in the box to the left of the group name. The color helps in recognizing a group while navigating through the software. Right-click to edit or clear groups.
Save only reflections that span images	With this checkbox enabled, a spot is only harvested if it is found on multiple contiguous frames.
[left arrow]	Go to the previous step in the unit cell determination process.
[right arrow]	Go to the next step in the unit cell determination process.
Finish	Finish the sequence of steps in the unit cell determination process, beginning with the current step. Perform any tasks that are necessary.
Harvest	Proceed with harvesting according to the options set in the preceding menu items.
Cancel	Leave Manual Mode and return to the initial Unit Cell Determination page, performing no operations and ignoring any changes made to menu items.

Index

Figure 5.8 — Index menu

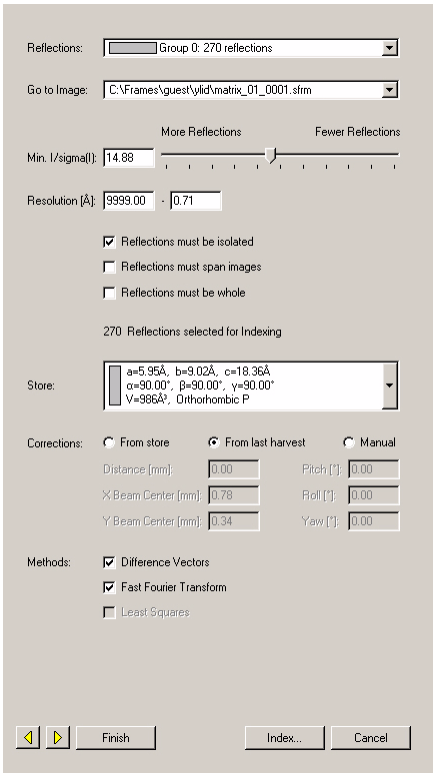


Table 5.3 — Index menu items

Menu Item	Function
Reflections:	Opens the Reflection Group Combo Box to allow you to select a certain group of reflections for indexing.
Go to Image:	Select a frame filename from this drop-down menu to display the frame in the Image Display Area.
Min I/sigma(I):	Adjust the criterion for indexing a reflection based on its integrated intensity versus its standard deviation. Slide the slider between “More Spots” and “Fewer Spots” to vary the minimum I/sigma(I) needed for a reflection to qualify for indexing.
Resolution (Å):	Set the resolution range from which spots will be used to index the unit cell.
Reflections must be isolated	If this checkbox is active, overlapping spots will not be used for indexing.
Reflections must span images	If this checkbox is active, only spots which registered on at least two adjacent images will be used for indexing.
Reflections must be whole	If this checkbox is active, spots that have only partially registered on the first and last images of the range from which they are harvested will not be used for indexing.

Menu Item	Function
Store (Unit Cell Combo Box):	The Unit Cell Combo Box shows a list of unit cells to choose from. For each unit cell, the unit cell parameters are displayed, as is the unit cell volume and, optionally, the Bravais lattice type. If, instead of the unit cell parameters, an entry is labeled empty, this indicates an unassigned entry. Choosing the empty entry creates a new unit cell instead of replacing the current one. A color is associated with each unit cell. The color is displayed in the box to the left of the unit cell. The color helps in recognizing a unit cell while navigating through the software. It is also used in the image display to color the overlay and indicate the unit cell that was used to calculate the overlay. Right-click to edit or clear unit cells.
Corrections:	Select corrections to detector parameters. Corrections are available from the stored parameters (i.e., from the Sample Database), from the last harvest, or from manual input.
Methods:	Select methods employed in indexing: difference vectors, fast fourier transform, or least squares.
[left arrow]	Go to the previous step in the unit cell determination process.
[right arrow]	Go to the next step in the unit cell determination process.
Finish	Finish the sequence of steps in the unit cell determination process, beginning with the current step. Perform any tasks that are necessary.
Index	Proceed with indexing according to the options set in the preceding menu items.
Cancel	Leave Manual Mode and return to the initial Unit Cell Determination page, performing no operations and ignoring any changes made to menu items.

Bravais

Figure 5.9 — Bravais menu

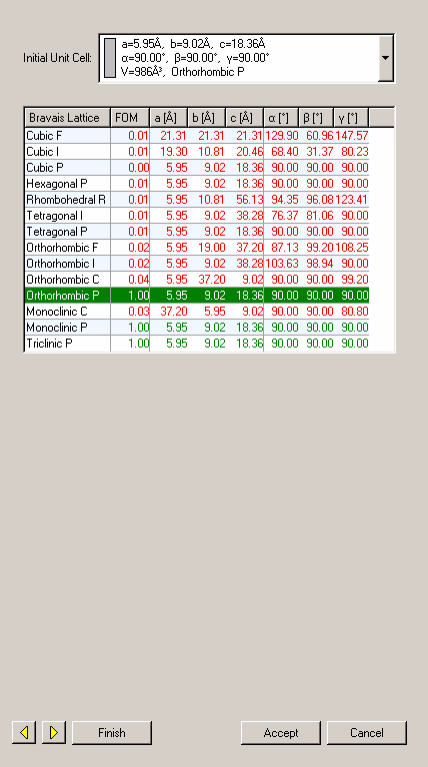


Table 5.4 — Bravais menu items

Menu Item	Function
Initial Unit Cell:	Use the Unit Cell Combo Box to select a unit cell to which to assign a Bravais lattice type.
Bravais Lattice:	This list shows the 14 Bravais lattice types for the selected unit cell parameters. For each entry, the list contains (from left to right) the Bravais lattice type, the figure of merit (ranging from 0.0 to 1.0, with 1.0 being perfect agreement), and the six unconstrained unit cell parameters for that Bravais lattice type. Bravais lattices that are in agreement with the unit cell are displayed in green, while those that do not are displayed in red. The most likely Bravais lattice type is chosen automatically. If necessary, you may override the program's decision by clicking on a different entry.
[left arrow]	Go to the previous step in the unit cell determination process.
[right arrow]	Go to the next step in the unit cell determination process.
Finish	Finish the sequence of steps in the unit cell determination process, beginning with the current step. Perform any tasks that are necessary.
Accept	Accept the choice of Bravais lattice type given in the menu items above.
Cancel	Leave Manual Mode and return to the initial Unit Cell Determination page, performing no operations and ignoring any changes made to menu items.

Refine

Figure 5.10 —Refine menu

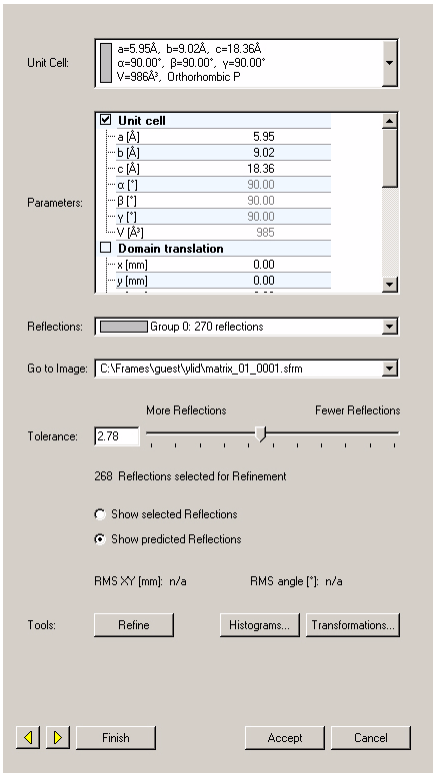


Table 5.5 — Refine menu items

Menu Item	Function
Unit Cell:	Use the Unit Cell Combo Box to select a unit cell to refine.
Parameters:	Within the Parameters window, several areas are available for refinement: Unit Cell, Domain Translation, Domain Orientation, Detector Translation, Beam Center, Detector Orientation, and Goniometer Zeros. By activating or deactivating the checkbox on the left, the parameters in this group are selected for refinement or are constrained to their current values during the refinement.
Reflections:	Use the Reflection Group Combo Box to the select a group of reflections for refinement.
Go to Image:	Select a frame filename from this drop-down menu to display the frame in the Image Display Area.
Tolerance:	Upon startup, the Refine dialog determines a useful value for the tolerance. This may take a few seconds depending on the number of reflections.
Show selected reflections	In the Image Display Area, reflections selected for refinement are marked with circles.
Show predicted reflections	In the Image Display Area, predicted reflections are marked with circles.
RMS XY [mm], angle [°]:	The root mean square of the deviation between observed and predicted spot positions in the XY image plane and along the trajectory of the spot while it passed through the Ewald sphere.
Tools:	Refine – Refine the unit cell using the selected parameters. Histograms... – Displays histograms of reflections' variances in H, K, L, detector X, detector Y, and rotation angle. Transformations... – Opens a dialog for transforming the unit cell.

Menu Item	Function
[left arrow]	Go to the previous step in the unit cell determination process.
[right arrow]	Go to the next step in the unit cell determination process.
Finish	Finish the sequence of steps in the unit cell determination process, beginning with the current step. Perform any tasks that are necessary.
Accept	Accept the options set in the preceding menu items, and proceed with a single cycle of least-squares refinement. The Refine dialog has a built-in safety which checks whether enough reflections are available for the refinement. If the number of reflections is critically low, a warning is displayed with the option to override.
Cancel	Leave Manual Mode and return to the initial Unit Cell Determination page, performing no operations and ignoring any changes made to menu items.

5.4 Determining the Data Collection Strategy

APEX2 includes powerful algorithms for determining flexible, efficient data collection strategies for your instrument.

These features are available in two modules, depending on your preference:

- The Nonius Collect-style Data Collection Strategy module (Section 5.4.1); and
- The Bruker Data Collection Strategy Module (Section 5.4.2).

5.4.1 Using the Collect-Style Data Collection Strategy Module

Start the Data Collection Strategy Module

1. In the Task Bar's "Collect" category, click the **Data Collection Strategy** icon .

   The Data Collection Strategy module opens.

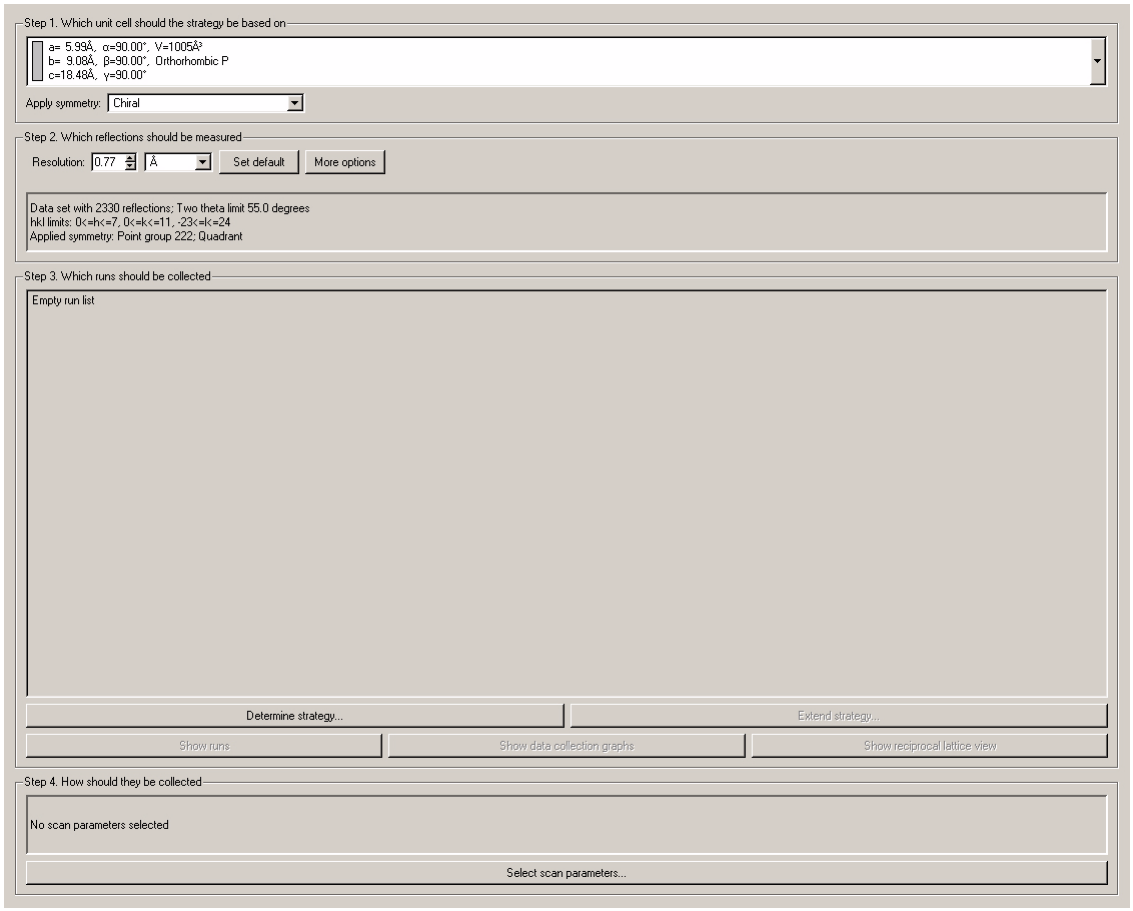
Choose the Unit Cell and Symmetry

1. In the "Step 1" area, choose the desired unit cell from the drop-down menu.
2. Use the "Apply symmetry:" drop-down menu to choose the symmetry.

   Choose to treat Bijvoet pairs as equivalent (**Centrosymmetric**) or inequivalent (**Chiral**), or choose a specific point-group symmetry based on the metric symmetry of the unit cell.

   A full sphere may be collected using a setting of **1**.

Figure 5.11 — Initial view with unit cell and symmetry chosen



Choose the Reflections to be Measured

1. In the “Step 2” area, choose an appropriate resolution limit.
2. If desired, click the **More options** button to open an expanded menu that allows measurement of data based on an input reflection file, collection based on a list of previously-collected data, or collection based on the orientation matrix (the default setting is **Unique set**).

Table 5.6 — **More options** function descriptions

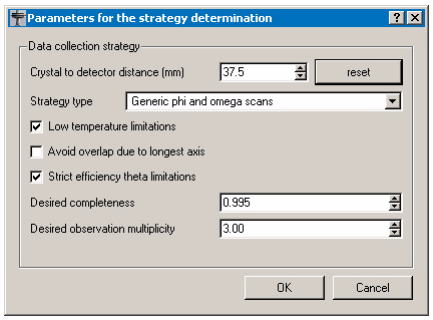
Function	Description
Unique set	Collects data based on the orientation matrix and resolution limit provided.
From file/Reflection file:	Calculates a strategy to collect the reflections contained in an existing .hkl file.
From file/Already measured:	Calculates strategy to collect reflections missing between input file and input resolution limit.

Determine the Runs to be Collected

1. In the “Step 3” area, click the **Determine Strategy...** button.

   The “Parameters for the strategy determination” window opens.

Figure 5.12 —“Parameters for the strategy determination” window



2. Choose the strategy determination options:
   2.1. Set the crystal-to-detector distance.
   2.2. Choose the strategy type (phi and omega scans, or simply omega scans).

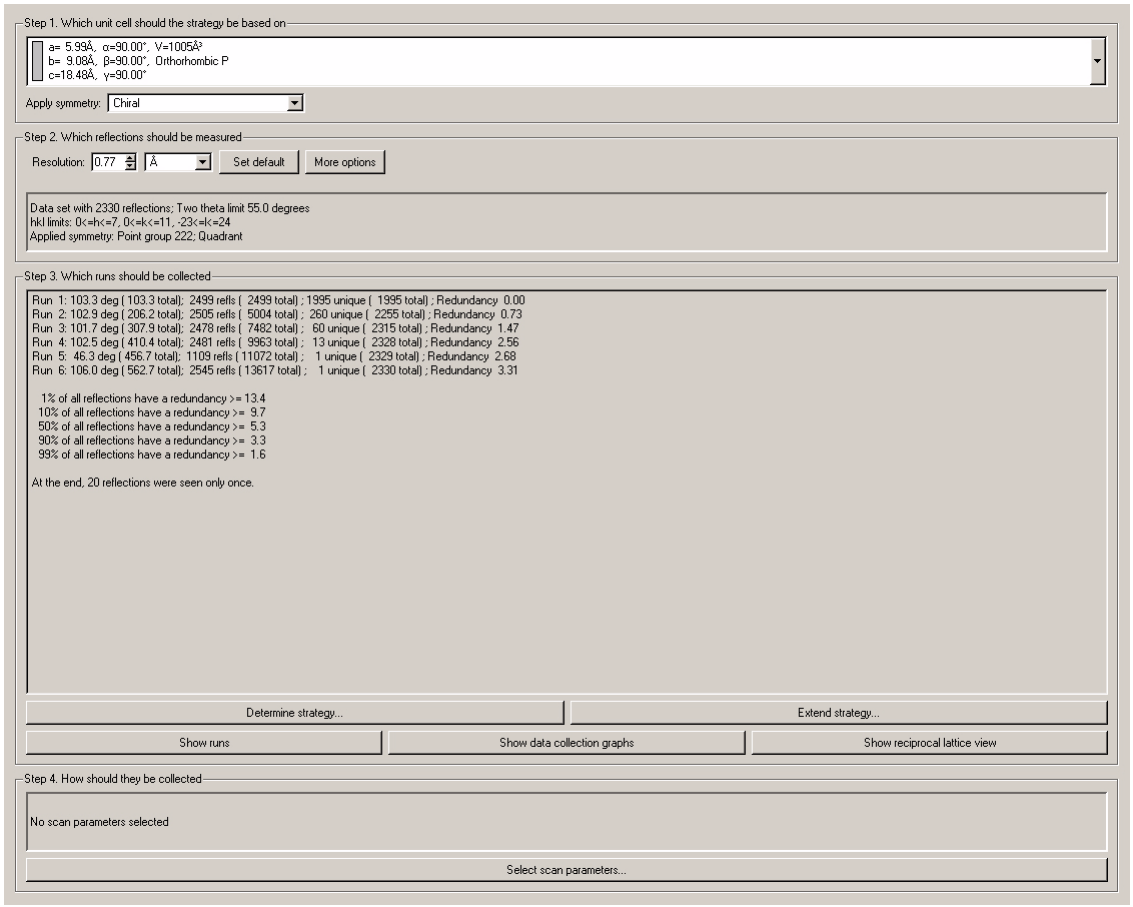
   Kappa goniometers also offer options for collecting Friedel pairs in the same frame (with omega scans for those that cannot be collected on the same frame) and for two 360° phi scans in reverse-beam geometry.
   2.3. Choose whether to avoid space occupied by the low-temperature device.
   2.4. Choose whether to avoid overlapping reflections by orienting the unit cell’s longest axis parallel to the detector.
   2.5. Choose whether to limit the swing of 2theta to improve efficiency (this may limit the ability to acquire all data for triclinic settings).
   2.6. Set the desired completeness and multiplicity (i.e., redundancy).

3. Click **OK**.

   The module computes the data collection strategy.

   After the calculation, a summary appears showing the sweep for each run, the number of reflections to be collected, the number of unique reflections to be collected, and the redundancy achieved (this is cumulative). Following this is a summary of the reflection distribution with respect to multiplicity. For example, redundancy at 90% is the lowest redundancy measured for 90% of the unique reflections.

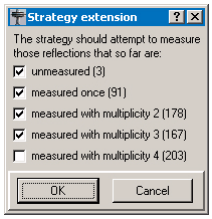
Figure 5.13 —Runs found



4. If desired, click **Extend strategy...** to specify collection of additional data based on multiplicity.

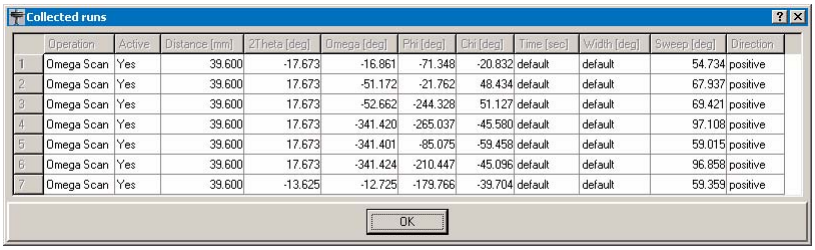
NOTE: Extending the strategy will increase data collection time.

Figure 5.14 —“Strategy extension” window



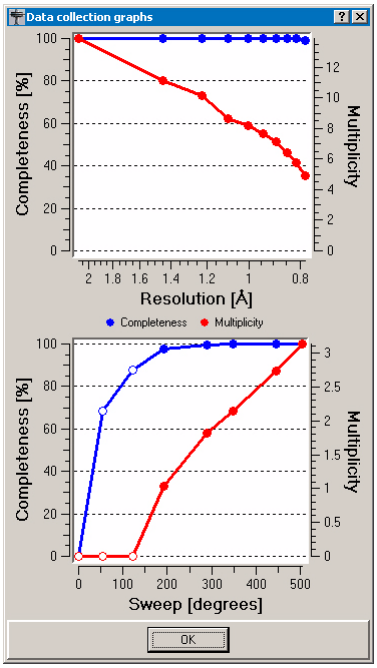
5. If desired, click **Show runs** to see the scan parameters for the calculated runs.

Figure 5.15 —"Collected runs" window



	Operation	Active	Distance [mm]	2Theta [deg]	Omega [deg]	Phi [deg]	Chi [deg]	Time [sec]	Width [deg]	Sweep [deg]	Direction
1	Omega Scan	Yes	39.600	-17.673	-16.861	-71.348	-20.832	default	default	54.734	positive
2	Omega Scan	Yes	39.600	17.673	-51.172	-21.762	48.434	default	default	67.937	positive
3	Omega Scan	Yes	39.600	17.673	-52.662	-244.328	51.127	default	default	69.421	positive
4	Omega Scan	Yes	39.600	17.673	-341.420	-265.037	-45.580	default	default	97.108	positive
5	Omega Scan	Yes	39.600	17.673	-341.401	-85.075	-59.458	default	default	59.015	positive
6	Omega Scan	Yes	39.600	17.673	-341.424	-210.447	-45.096	default	default	96.858	positive
7	Omega Scan	Yes	39.600	-13.625	-12.725	-179.766	-39.704	default	default	59.359	positive

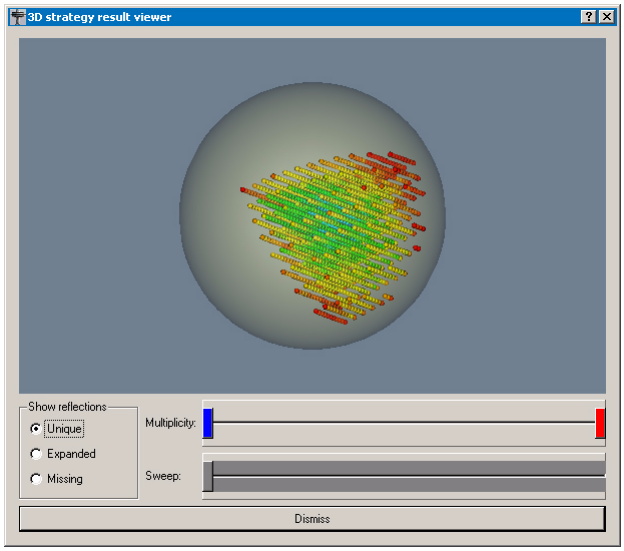
6. If desired, click **Show data collection graphs** to display graphs showing completeness and multiplicity as a function of resolution (upper graph), and as a function of the data collection sweep (lower graph).



7. If desired, click **Show reciprocal lattice view** to display the strategy in reciprocal space.

   Options are available to display the unique data to be collected, the data expanded across the sphere, or only the missing reflections.
   The **Multiplicity** sliders apply a lower-redundancy cutoff (left slider), or a higher-redundancy cutoff (right slider) to help visualize the data collection. The **Sweep** slider can be used to examine the data collection coverage with respect to experiment time.

Figure 5.16 —"3D strategy result viewer" window

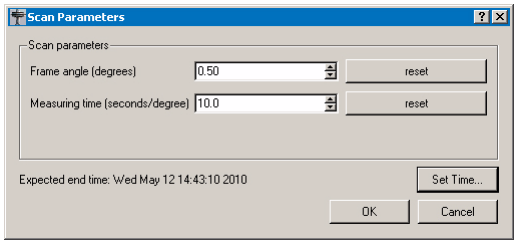


Set the Scan Parameters

1. In the "Step 4" area, click the **Select scan parameters...** button.

   The "Scan Parameters" window opens.

Figure 5.17 —"Scan Parameters" window



2. Set the scan parameters:
   2.1. Set the desired scan width (i.e., frame angle) based on the crystal's mosaicity.
   2.2. Set the scan time, in seconds per degree.
   2.3. If desired, click the **Set Time...** button to open a dialog that allows you to set the finish time (this adjusts the measuring time to best fill the time allowed).

3. Click **OK**.

   The "Scan Parameters" window closes. Note that the elapsed time and time of day have been added to the run list in the "Step 3" area.

The strategy calculation is now complete. The module may be closed, and the runs may be imported to the Experiment module. Open the Experiment module, and click **Append Strategy** to move the calculated runs into the experiment's table of operations.

5.4.2 Using the Data Collection Strategy Module

Start the Data Collection Strategy Module

1. In the Task Bar's "Collect" category, click the **Data Collection Strategy** icon .

   The Data Collection Strategy module opens.

Set Up the Strategy Calculation

NOTE: The Data Collection Strategy module uses information from the Determine Unit Cell module to set defaults. However, you can modify the suggested values.

1. Set the data collection distance.

   For SMART APEX II systems, this should be set to the actual detector distance. For KAPPA APEX II systems, the distance is variable and will default to the shortest reasonable distance. For the APEX II detector, the distance in millimeters should generally be about the same as the longest cell dimension in angstroms. Typically, distances ranging from 35 mm to 45 mm are reasonable.
2. Set the exposure time, and press [Enter].
3. Click **Same** to set all of the times to be the same.

NOTE: If the "Same" feature is not chosen, the times for resolution shells can be set to collect high-angle data with longer exposures than inner shell data.

4. Set the desired resolution (0.75Å is a reasonable value).
5. Check the other values (Laue class, Lattice, etc.).
6. Each time a value is changed, the Data Collection Strategy module recalculates the statistics for the runs. The results are displayed in the "Current" column.

NOTE: The rescanning of runs can be interrupted by pressing [Esc]. No statistics will be displayed.

Refine the Strategy

Before proceeding with the experiment, it is necessary to optimize the data collection strategy for your desired experimental parameters (e.g., experiment duration or redundancy).

1. Below the “Target” and “Priority” columns is a pull-down menu with several different strategies. Choose the one that best meets the needs of the experiment.

   The “Target” column changes to reflect your choice.

2. Click in the “Execute:” field, and choose **Refine Strategy**.

Figure 5.18 —Click **Refine Strategy**

   The strategy begins to refine, approaching the target Completeness, Redundancy, and Time.

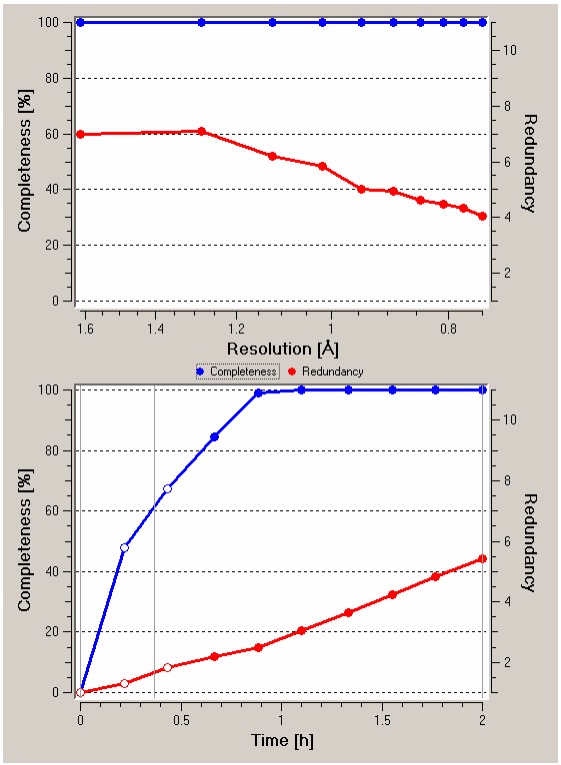
3. When you are satisfied with the Completeness, Redundancy, and Time displayed in the “Current” column (whether the module is finished refining or not), click **Stop**.

   It is neither necessary nor advisable to wait until the refinement reaches 100%.

NOTE: When refining the strategy, the objective is to get good completion (98% or better) with high redundancy in a reasonable amount of time. When the Data Collection Strategy module is first started, it will tell you the Completeness, Redundancy, and Time for all of the available runs.

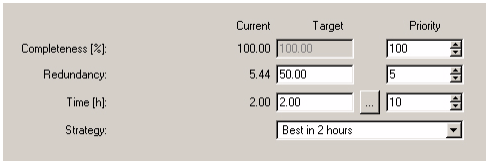
NOTE: It is almost never necessary to let the Data Collection Strategy module run to completion. It may be stopped when the parameters approach the desired values.

Figure 5.19 —Completeness and redundancy chart



NOTE: Time estimates are approximate. They depend on the number of rescans, general instrument overhead, backlash compensation, etc. If estimated times are consistently longer or shorter, it may be advisable to modify the COSMO hardware profile (see DOC-M86-EXX112 COSMATIC Fixed-Chi Geometry Hardware Profile Procedure or DOC-M86-EXX113 COSMATIC Kappa Geometry Hardware Profile Procedure as applicable).

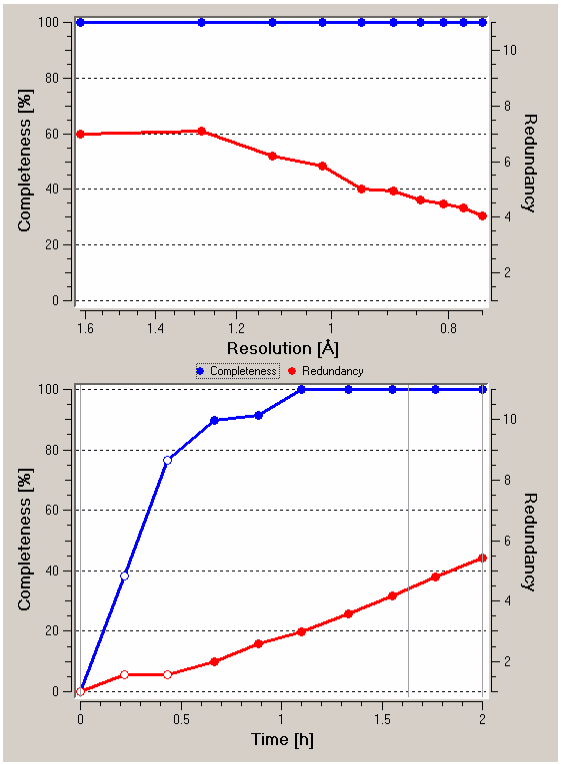
Figure 5.20 —Strategy status and priority control



4. Click in the “Execute:” field, and choose **Sort Runs for Completeness**.

Figure 5.21 —Sort runs for completeness

Figure 5.22 —Completeness and redundancy after sorting for completeness



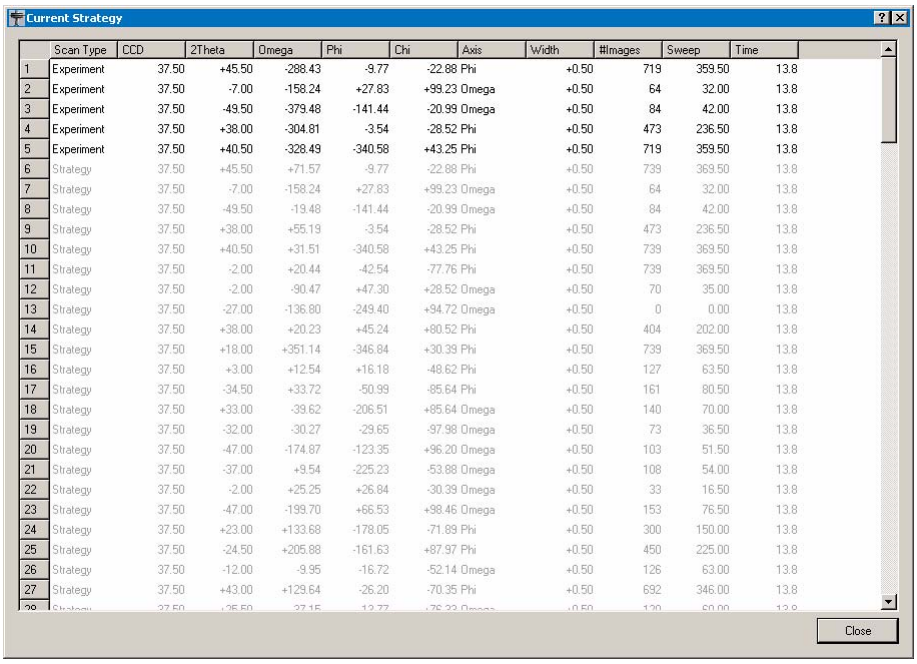
NOTE: If it becomes necessary to start the refinement process over, change the distance slightly (e.g., by 0.02) and press [Enter]. The Data Collection Strategy module will reload its list of possible runs.

Working with the Current Strategy

1. To look at the actual runs output by the Data Collection Strategy Module, choose **View > Detailed Strategy**.

   A window opens showing the runs to be collected (see Figure 5.23).

Figure 5.23 —Current Strategy window



	Scan Type	CCD	2Theta	Omega	Phi	Chi	Axis	Width	#Images	Sweep	Time
1	Experiment	37.50	+45.50	-288.43	-9.77	-22.88	Phi	+0.50	719	359.50	13.8
2	Experiment	37.50	-7.00	-158.24	+27.83	+99.23	Omega	+0.50	64	32.00	13.8
3	Experiment	37.50	-49.50	-379.48	-141.44	-20.99	Omega	+0.50	84	42.00	13.8
4	Experiment	37.50	+38.00	-304.81	-3.54	-28.52	Phi	+0.50	473	236.50	13.8
5	Experiment	37.50	+40.50	-328.49	-340.58	+43.25	Phi	+0.50	719	359.50	13.8
6	Strategy	37.50	+45.50	+71.57	-9.77	-22.88	Phi	+0.50	739	369.50	13.8
7	Strategy	37.50	-7.00	-158.24	+27.83	+99.23	Omega	+0.50	64	32.00	13.8
8	Strategy	37.50	-49.50	-19.48	-141.44	-20.99	Omega	+0.50	84	42.00	13.8
9	Strategy	37.50	+38.00	+55.19	-3.54	-28.52	Phi	+0.50	473	236.50	13.8
10	Strategy	37.50	+40.50	+31.51	-340.58	+43.25	Phi	+0.50	739	369.50	13.8
11	Strategy	37.50	-2.00	+20.44	-42.54	-77.76	Phi	+0.50	739	369.50	13.8
12	Strategy	37.50	-2.00	-90.47	+47.30	+28.52	Omega	+0.50	70	35.00	13.8
13	Strategy	37.50	-27.00	-136.80	-249.40	+94.72	Omega	+0.50	0	0.00	13.8
14	Strategy	37.50	+38.00	+20.23	+45.24	+80.52	Phi	+0.50	404	202.00	13.8
15	Strategy	37.50	+18.00	+351.14	-346.84	+30.39	Phi	+0.50	739	369.50	13.8
16	Strategy	37.50	+3.00	+12.54	+16.18	-48.62	Phi	+0.50	127	63.50	13.8
17	Strategy	37.50	-34.50	+33.72	-50.99	-85.64	Phi	+0.50	161	80.50	13.8
18	Strategy	37.50	+33.00	-39.62	-206.51	+85.64	Omega	+0.50	140	70.00	13.8
19	Strategy	37.50	-32.00	-30.27	-29.65	-97.98	Omega	+0.50	73	36.50	13.8
20	Strategy	37.50	-47.00	-174.87	-123.35	+96.20	Omega	+0.50	103	51.50	13.8
21	Strategy	37.50	-37.00	+9.54	-225.23	-53.88	Omega	+0.50	108	54.00	13.8
22	Strategy	37.50	-2.00	+25.25	+26.84	-30.39	Omega	+0.50	33	16.50	13.8
23	Strategy	37.50	-47.00	-199.70	+66.53	+98.46	Omega	+0.50	153	76.50	13.8
24	Strategy	37.50	+23.00	+133.68	-178.05	-71.89	Phi	+0.50	300	150.00	13.8
25	Strategy	37.50	-24.50	+205.88	-161.63	+87.97	Phi	+0.50	450	225.00	13.8
26	Strategy	37.50	-12.00	-9.95	-16.72	-52.14	Omega	+0.50	126	63.00	13.8
27	Strategy	37.50	+43.00	+129.64	-26.20	-70.35	Phi	+0.50	692	346.00	13.8

Run Properties and Behaviors

Each line in the “Current Strategy” window represents one run. It is specified by:

- Origin
- Crystal-to-detector distance
- Angular positions of the goniometer axes
- Name of the scan axis
- Image width
- Number of Images
- Total Sweep
- Exposure time + overhead

Each run is displayed in a color that indicates its behavior during the strategy refinement:

Table 5.7 — Run colors and behaviors

Run Color	Behavior
Black	Permanent runs that are always active during the refinement process. They are not altered during the refinement. This option is intended for runs that have already been collected and therefore definitively contribute to the data set.
Red	“As-is” runs that, unlike the permanent runs, can be disabled by the refinement algorithm. However, the configuration of such a run remains unchanged (e.g., the scan range will not change).
Blue	Runs that can change their scan range during the course of the refinement. They are therefore called “partial runs”. This is the default option for all runs from the basis set, because it gives the refinement algorithm the highest flexibility in fulfilling the criteria for completeness, redundancy, and total data collection time.
Gray	Disabled runs that are not taken into account at any time during the strategy refinement.

Change the Behavior of a Run

To change the behavior of a run:

1. Place the Data Collection Strategy algorithm in idle mode (i.e., make sure that it is not calculating the reflection list, refining a strategy, or sorting the run list).
2. Select the run or runs you wish to change by clicking or dragging the mouse inside the “Current Strategy” window.
3. Right-click inside the “Current Strategy” window.

   A right-click menu opens.
4. In the right-click menu, choose the desired behavior (permanent, as-is, partial, or disabled). The “Current Strategy” window and the contents of the Main Window will then be updated.

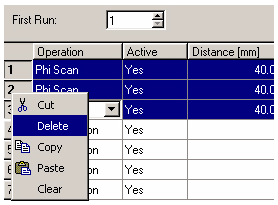
5.5 Run the Experiment

1. In the Task Bar's "Collect" category, click the **Experiment** icon .

   The Experiment module opens, showing a table of operations to be performed.

2. If the list of operations contains orientation matrix runs, select and delete them.

Figure 5.24 —Deleting matrix runs

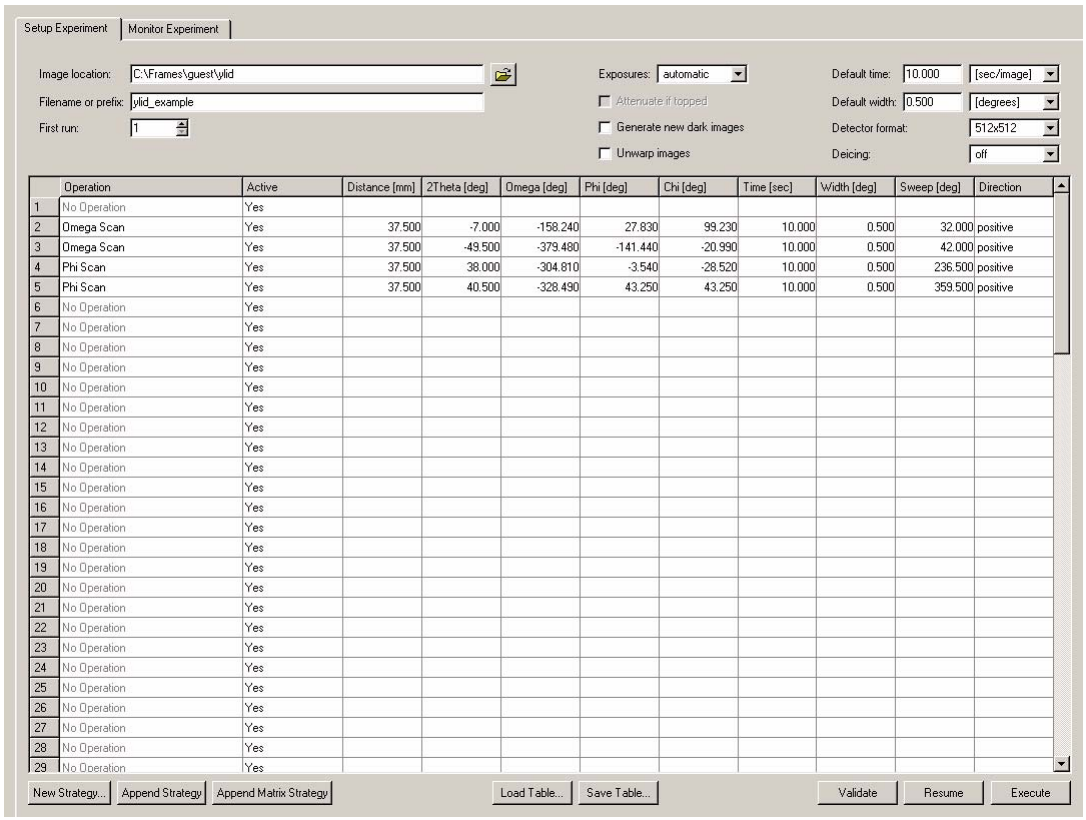


3. Click **Append Strategy**.

   The runs found by the Strategy Determination Module appear in the list of operations.

   The software changes the filename to the name of the current sample.

Figure 5.25 —List of operations with strategy appended

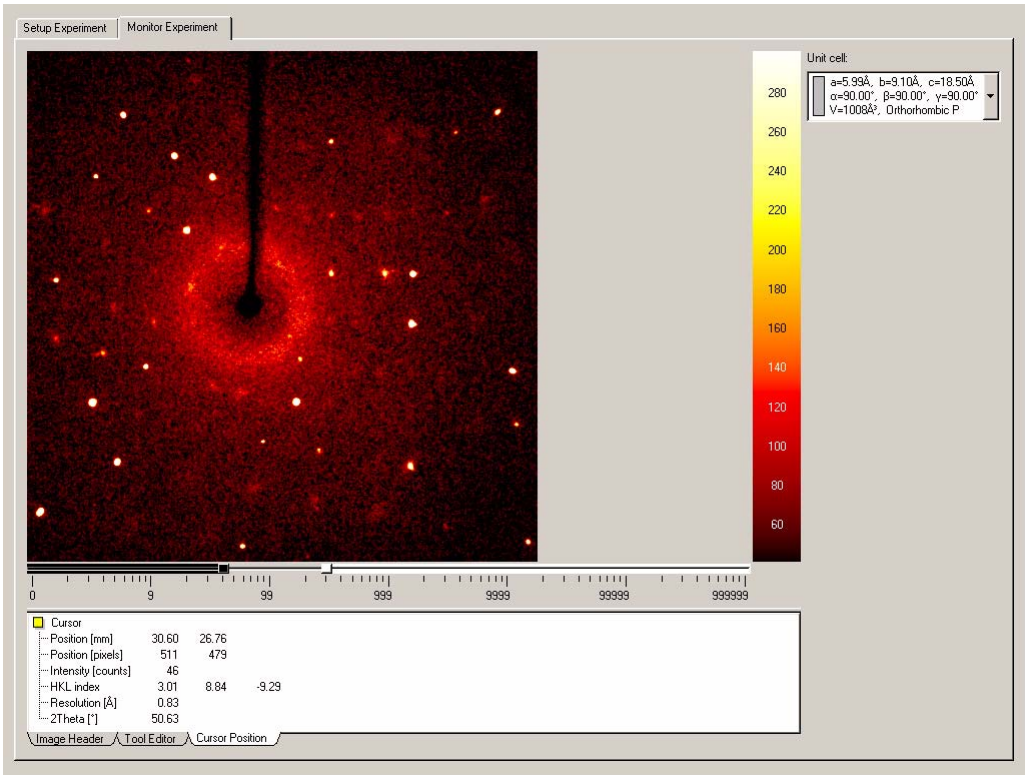


4. To begin the experiment, click **Execute**.

   The "Monitor Experiment" tab opens, and diffraction images begin to appear. This may take a minute or two if new dark current corrections are being collected, or if the generator is being ramped up from a low-power state.

NOTE: If resuming after a data collection interruption, the system will automatically skip images that were previously collected with matching angles and generator settings. Otherwise, the system will ask if you want to overwrite the images.

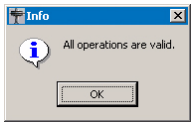
Figure 5.26 —"Monitor Experiment" tab



5.5.1 User-Defined Runs and Settings

You can load and save your own experiments (in *.exp format) by using the **Load Table...** and **Save Table...** buttons at the bottom of the “Setup Experiment” tab.
At the top of the “Setup Experiment” tab are controls for data collection. Usually, the default values are correct. For data collection times of less than five seconds, correlation can usually be turned off. If new dark frames are required, BIS will automatically collect them. Activating the “Generate New Darks” checkbox forces the collection of darks before every run. In Figure 5.25, the time and width are explicitly set for each run, so changing the default width and time will have no effect. If the explicit time or width for a run is deleted so that the box is empty, the word `default` appears and the default values at the top right will be used.
If you choose to enter your own runs, click **Validate** to test the strategy for illegal goniometer movements. If all movements are valid, the “All operations are valid” window will appear (Figure 5.27).

Figure 5.27 —“All operations are valid” window



5.5.2 Exiting During Data Collection

If desired, you can exit APEX2 after you begin data collection:

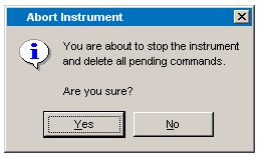
- In a single-computer setup, BIS must be left running.
- In a dual-computer setup, the Server computer must be left on. If communications are lost between the Client and the Server, frames will be stored on the Server. Typically, they will be in the directory C:\frames\. They should be copied into the correct project directory before proceeding.

5.5.3 Abort the Experiment if Necessary

1. To abort the data collection, choose **Instrument > Abort...** or click the **Stop** button in the Tool Icon Bar.

   You are prompted to confirm that you wish to abort the data collection.

Figure 5.28 —“Abort Instrument” window



2. Click **Yes** to abort data collection.

NOTE: If resuming after a data collection interruption, the system will automatically skip images that were previously collected with matching angles and generator settings. Otherwise, the system will prompt you to confirm that you wish to overwrite the images.

6 Data Integration and Scaling

Before the data can be used to solve and refine the crystal structure, it is necessary to convert the information recorded on the frames to a set of integrated and scaled intensities.

When setting up the integration, the Integrate Images module creates a subdirectory named “\work” and assigns an output filename for each run’s integration results. These files are in ASCII form, which can be input into XScale and XPREP to produce a scaled intensity file. The SAINT Software Reference Manual DOC-269-0175XX provides a detailed description of the process of integration along with the various parameters involved.

A detailed description of the SAINT file format is available in BIS’ online help.

It is possible to integrate while collecting data.

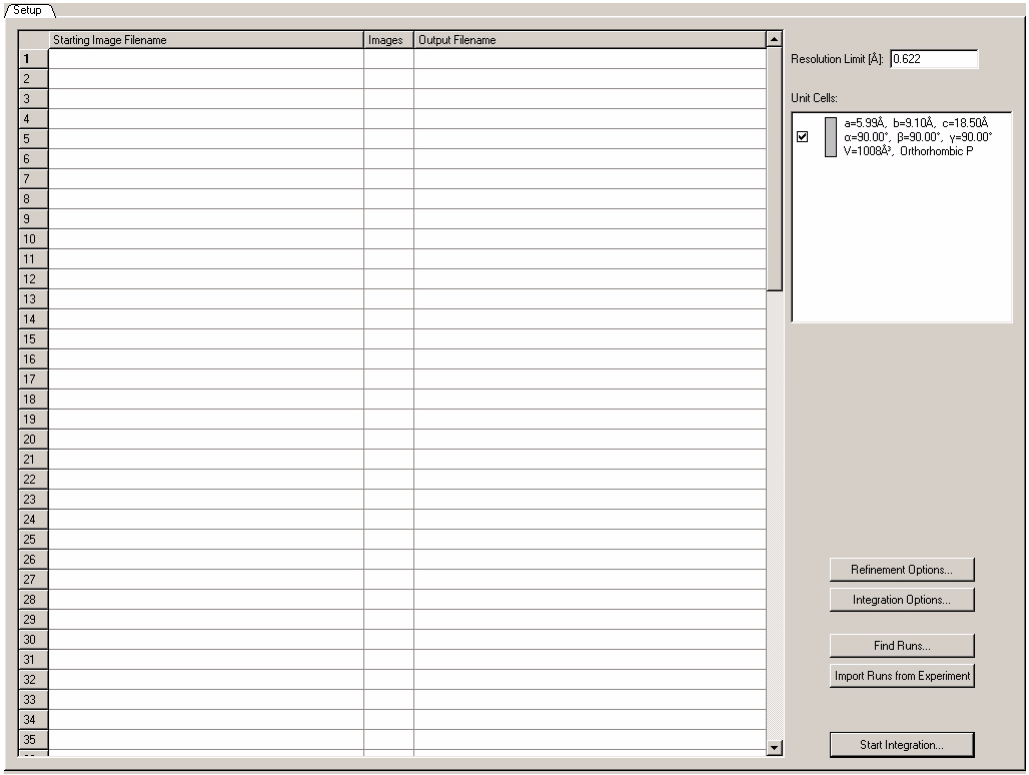
Corrections for crystal decay and X-ray absorption can also be made, and data from different runs and different crystals can be merged and scaled. A detailed statistical analysis of the data is produced in tabular and graphical form.

6.1 Start the Integrate Images Module

1. In the Task Bar's "Integrate" category, click the **Integrate Images** icon .

   The Integrate Images module opens, and the orientation matrix and Bravais information are loaded from the database.

Figure 6.1 — Integrate Images module: initial view



6.2 Set the Resolution Limit

The "Resolution Limit (Å):" field sets the upper (i.e., better) resolution limit to which the data will be integrated. The module suggests a resolution limit, but this value is editable.

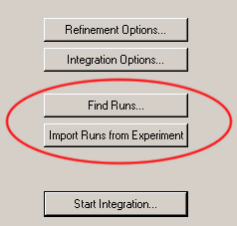
1. Check the suggested resolution limit. If you want a limit other than that suggested, enter it in the field.

Figure 6.2 — "Resolution Limit (Å):" field

6.3 Select Runs for Integration

The module's right-hand side has two buttons for defining the data collection runs to be integrated: **Find Runs...** and **Import Runs from Experiment**.

Figure 6.3 — **Find Runs** and **Import Runs from Experiment** buttons



6.3.1 Browse for Runs

The **Find Runs...** button is used to browse to the set of runs to be integrated. This button is normally used when the data collection is finished.

1. Click **Find Runs...**.

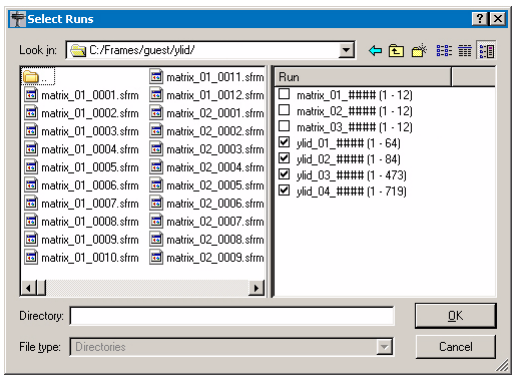
   The "Select Runs" window (Figure 6.4) opens.

2. Usually, the "Select Runs" window has the correct directory and base name as the defaults. If these are not correct, use the **Browse** button to find the runs for the experiment of interest.

NOTE: To integrate images from a CD or DVD, browse to the device and find the runs to be integrated. The module automatically writes the result in the "\work" directory for the current project.

3. The window pane on the right-hand side displays all of the runs available in the current directory. Any combination of runs may be selected by simply activating the checkboxes next to the groups.

Figure 6.4 — "Select Runs" window



4. Click **OK**.

   The module populates the list of runs to be integrated with the selected runs.

6.3.2 Import Runs from Experiment

The **Import Runs from Experiment** button determines the runs to be integrated from the experiment that has just been submitted. Using this button allows you to start integration while the data is still being collected.

NOTE: When integrating while collecting data, the SAINT integration module will integrate all of the data currently measured and then wait for the next image, integrate that image, wait for the next, etc.

1. Click **Import Runs from Experiment**.

   The module populates the list of runs to be integrated with the runs from the experiment.

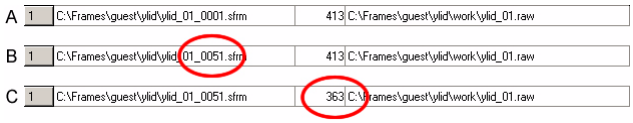
6.3.3 Modifying Integration Run Lists

By editing the fields in the run list, the starting image name and output filename can be changed. You can use standard clipboard tools (e.g., Copy, Paste) by selecting a run entry and right-clicking on the entry number. In the way, it is also possible to count the number of frames available for each run.

Figure 6.5 shows a run list entry being modified. (A) shows the original run information. (B) shows the run with the starting image number changed to 51. (C) shows the number of frames to process changed to 111.

Double-click any of the fields in this list to open the value for editing.

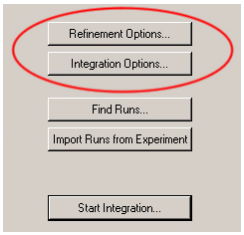
Figure 6.5 — Example: manually editing a run list



6.4 Set the Refinement Options

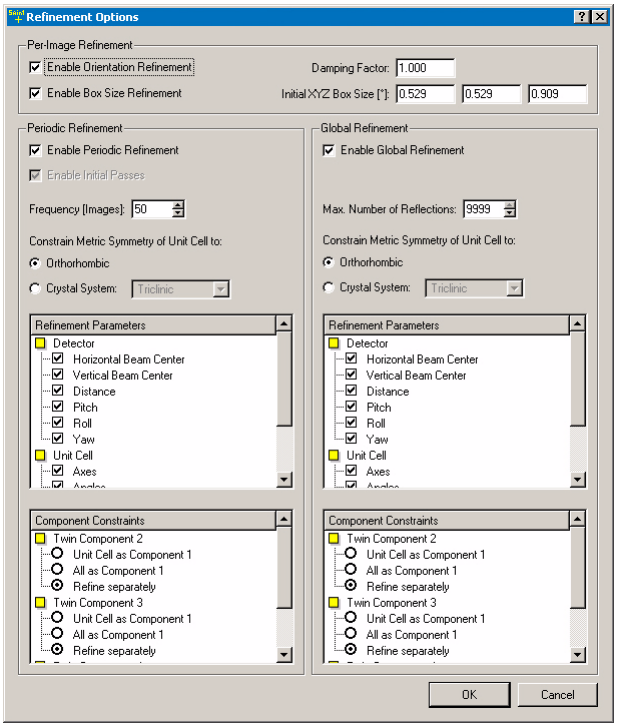
1. Click the **Refinement Options...** button in the lower right-hand corner of the module.

Figure 6.6 — Refinement and Integration Options buttons



The default values are generally very good. There is seldom any need to change the values in the “Refinement Options” window.

Figure 6.7 — “Refinement Options” window



NOTE: To change the default values that appear in the “Refinement Options” window, create a text file C:\saint.ini. Copy the sections and parameters you want to change from C:\bn\src\interface\saint.ini into C:\saint.ini (do not change C:\bn\src\interface\saint.ini).

2. Click **OK** to close the window.

6.4.1 Refinement Options Controls

Table 6.1 — Refinement options

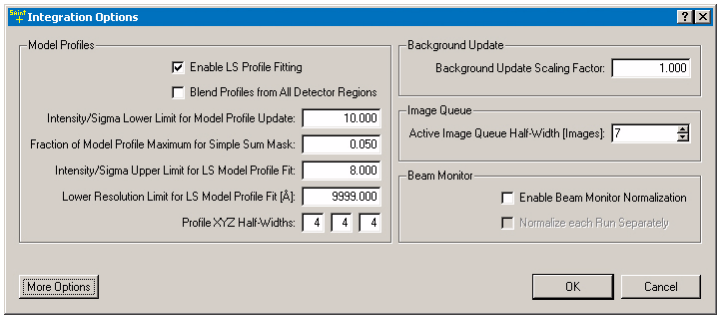
Option		Usage
Per-image refinement	Enable Orientation Refinement	Updates the crystal and detector parameters on a per-frame basis.
	Enable Box Size Refinement	Allows the program to refine the size of the box used to determine the model profiles. This can be very helpful if the spot shapes change as the crystal is rotated.
	Damping Factor	Varies the number of frames used in per-frame refinement. Best left alone unless you are an advanced user.
	Initial XYZ Box Size	The module's estimate of the spot size in degrees. The program tries to come up with a good starting value, but sometimes the box is too small. This value is refined at the beginning of integration, so it usually doesn't need to be changed unless instructed by the module.
Periodic refinement	Enable Periodic Refinement	Tells the module to perform a full-matrix least-squares orientation matrix refinement after every user-defined number of frames.
	Frequency (Images)	Shows how often the Periodic Refinement takes place.
	Constrain Metric Symmetry	Defaults to the current space group constraints. Clicking on the **Crystal System** radio button and selecting a symmetry class can change this.
	Refinement Parameters	Designates which parameters are to be refined.
Global refinement	Enable Global Refinement	After integration and output of sorted reflection files for the individual runs and merged reflection file, the module performs a global unit cell least squares. It uses data stored during integration in a temporary file (###. _MA). This file contains information only for reflections above the strong-reflection threshold and is required because some information needed to perform such a refinement (for example, all four setting angles for every reflection) is not in the output reflection file.
	Max. Number of Reflections	The total number of reflections used for global refinement. The default works well.

6.5 Set the Integration Options

1. Click **Integration Options...** on the module's right-hand side.

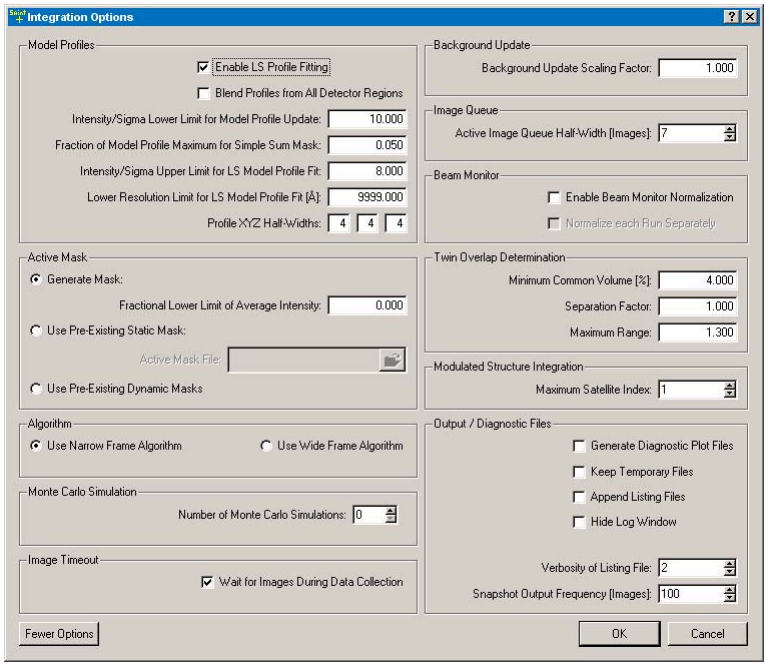
   The "Integration Options" window opens.

Figure 6.8 — "Integration Options" window



2. Click the **More Options** button to expand the window (see Figure 6.9).

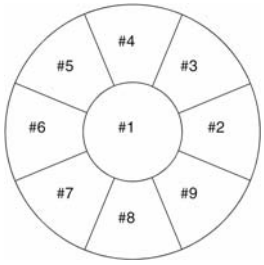
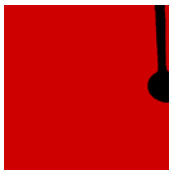
Figure 6.9 — "Integration Options" window (expanded)



3. Set the options as necessary for your data.
4. Click **OK**.

6.5.1 Integration Options Controls

Table 6.2 — Integration options

Option		Usage
Model Profiles	Enable LS Profile fitting	With this checkbox activated, the module will perform a per-frame least-squares fit between the observed reflections and the model profiles.
	Blend Profiles from All Detector Regions	During the integration, the model profile shape is determined separately for nine regions of the detector (see picture). Blending the model profiles results in less variation across the detector area, and may provide better statistics for regions where the reflections are very weak. 
	Intensity/Sigma lower limit	Reflections that meet this criteria are used to produce the model profiles. The default value of `10` is acceptable for most data sets. If the sample has a very weak scatter, lowering this value may produce a more representative profile.
Active Mask		The Active Mask is used to mask out areas that are covered by the beamstop, low-temperature device nozzle, or other obstructions. Typically a value of 0.7 is good here. The module will determine an active mask for each run. If this option is set, you should examine the active mask images that are written into the work directory with the names `<basename>_am_##.sfrm`. There is one for each run. These images can be opened with the image viewer. They should be blank except for an area in a different color showing the shape of the obstruction, typically the beamstop. 

Option		Usage
Algorithm	Use narrow frame algorithm	The wide-frame algorithm differs from the narrow-frame algorithm as follows: • Spot sizes in Z are estimated based on partiality statistics accumulated for those spots which span frames. • Orientation least squares uses both “fulls” and “partials”, with “fulls” given zero weight in Z. • Reflection profiles are collapsed in Z for purposes of correlation with model profiles and LS profile fitting. • Spots that collide in Z (i.e., spots at similar X and Y with overlapping intensity on a common frame) are recognized and excluded from output. Their Z centroids may occur on the same frame, or on different (and not necessarily adjacent) frames. Integration of wide-frame twin and modulated-structure data are both supported although not recommended, since problems with spot overlap will tend to increase. The wide-frame algorithm will usually give better results in cases where the frame width is substantially greater than the spot width. Acquisition of wide frames often degrades the signal-to-noise ratio, especially for weak reflections, and results in a higher number of overlapping spots. Narrow-frame data collection is therefore recommended, and will usually result in higher data quality.
	Use wide frame algorithm	
Image Timeout	Wait for images during data collection	The integration will wait for new images during data collection if this checkbox is activated.
Image Queue	Active Image Queue Half-Width (images)	The Image Queue sets the number of frames being used to determine profiles. For crystals with high mosaic spread (i.e., large, wide reflections), increasing the queue size can improve integration. The active image queue is the angular integration region. If the rocking curve of a reflection is smaller than the queue, the reflection is considered to be whole and is integrated. If not, the reflection is rejected as a partial. To increase or decrease the size of the queue, change the active image queue half-width. For example, if the half-width is 10, the image queue consists of 20 frames. If the rotation range for each frame were 0.5°, then the size of the queue would be 10°. Spots that are wider than 10° are rejected and are not written to the output file. Usually, the only reflections rejected are those with high Lorentz factors. To include more of these spots, increase the half-width for the image queue.
Output/Diagnostic Files	Generate Diagnostic Plot Files	If this option is set, the following diagnostic files will be generated: • XPREP reciprocal space plot file (*.EDI): This is a SHELXTL HKLF 4 file that can be viewed with XPREP’s Reciprocal Space Display. It indicates the disagreement among symmetry-related intensities as a function of 3D position in reciprocal space. • Scatter Plots (*.EXX, *.EXZ, *.EXI, *.EYY, *.EYZ, *.EZZ, *.EDX, *.EDY, *.EDZ,): Each of these files contains a scatter plot in PLOTSO format. you can use PLOTSO to view these files.

6.6 Start the Integration

1. Click the **Start Integration** button on the lower right-hand side of the module.

   Integration begins, and the SaintChart windows open for monitoring the integration.

   A file containing the unmerged raw intensities (.raw) is written for each run. If multiple runs are integrated, a merged intensity file is written as well. The filename of the merged file contains a “0m” for the run number.

   At the end of the integration run, the module carries out a global refinement of crystal and detector parameters, followed by a statistical analysis of the integrated data set. The results are reported in the text display.

6.6.1 Monitoring Integration Progress with SaintChart

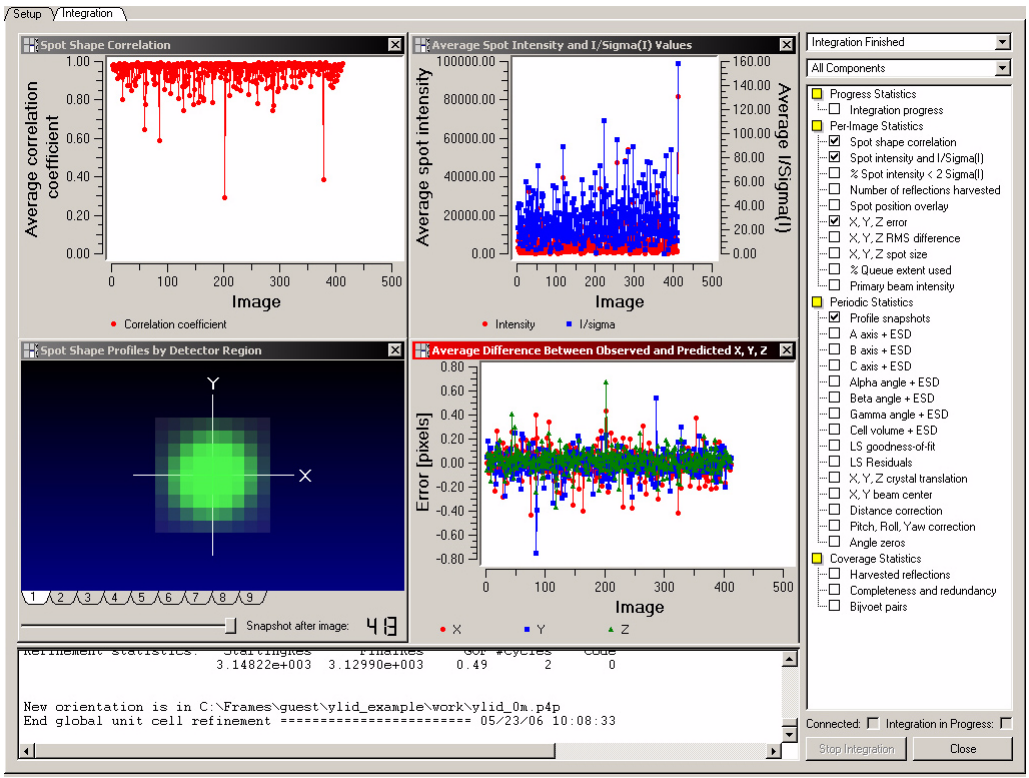
SaintChart is a powerful tool for monitoring the progress of the integration process and for graphically presenting the results of the integration process.

To the right of the image area are a series of checkboxes that select the displays. Activate or deactivate the checkboxes to add or remove displays.

Change the text area by clicking and dragging on the line between the text and image areas. You can also expand the windows to fill the available image area by clicking **Chart > Tile** in the Menu Bar.

After integration, the results are reported in the text display and a chart file (*.cht) is written to the work directory. You can open a chart file using **Chart > Open Chart File...** in the Menu Bar.

Figure 6.10 —SaintChart view



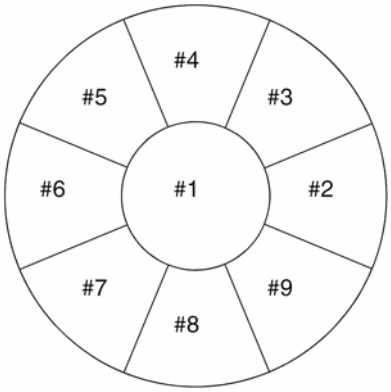
Average Difference

Errors in X, Y, and Z should be small (less than 1 pixel) and should not vary during the integration of a run. Large variations indicate problems with slipping crystals, misalignment of the instrument, or other problems.

Spot Shape Profiles by Detector Region

Spot shape profiles displays the model 3D reflection profiles in pixels for the nine regions of the detector. Holding the left mouse button down while moving the mouse enables the box to be rotated.

Figure 6.11 — Detector regions (viewed from the detector front)



In general, each 3D profile should be centered in the box but the spot shapes may change from region to region. Multiple or deformed 3D profiles indicate that your choice of initial box size or indexing may be incorrect. Split spot shapes may indicate diffraction from split crystals or twins.

Spot Shape Intensity and I/Sigma

Spot intensity and I/sigma plots the average intensity and average I/sigma (intensity divided by standard deviation) for the integrated reflections on a given image.

Spot Shape Correlation

The Spot Shape Correlation graph is the best indicator for a successful integration. It plots the degree of correlation between the measured 3D reflation profiles and the model 3D profiles computed from the strong spots.

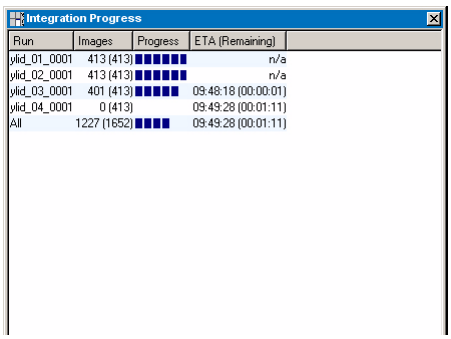
- For well-diffracting crystals with properly-chosen exposure times, the spot shape correlation should be in the range of 0.8 or better.
- For weak diffractors, the spot shape correlation will be lower but should be at least 0.4 if the exposure time is chosen correctly.
- Correlation factors below 0.4 indicate a serious problem with the indexing, the experimental setup, or both.

You can investigate any unusual features in the spot shape correlation by checking the images corresponding to the region of concern using the Movie Tool. To start the movie, right-click in the Image Display Area and choose **Movie**.

Integration Progress

Activate the **Integration Progress** checkbox to add the new display seen in Figure 6.12. When the integration is complete, the blue progress bars will be full and the remaining time will be zero.

Figure 6.12 —"Integration Progress" window



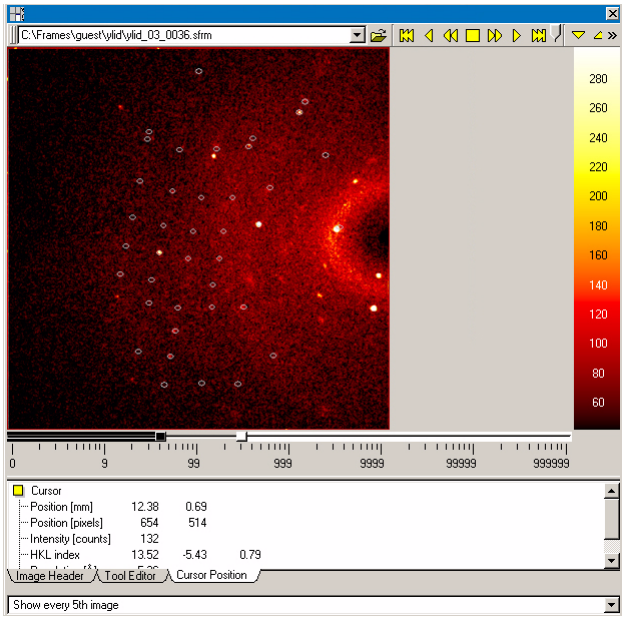
Spot Position Overlay

Activate the **Spot Position Overlay** checkbox (in the "Per-Image Statistics" category) to add a window for monitoring the progress and quality of the integration. Pull up on the Window Tool Bar to display the entire window.

In this full view, the "Integrated Spots" window looks much like the Image Viewer module. At the bottom of the window is an entry box that can be used to change the display frequency of the Integrated Spots images (the default is to display every fifth image).

The shape of the overlay represents the reflections' sizes and shapes determined during the integration.

Figure 6.13 —"Integrated Spots" window with spot position overlay



X, Y, Z Error

X, Y, Z error plots the average difference in pixels (X and Y) and frame number (Z) between the observed and predicted reflection positions on a given frame. These parameters are useful indicators of the module's ability to track reflections.

6.7 Examine the Integration Results

After integration, examine this text critically. In particular, look at the Overall R_{sym}, the Coverage Statistics, and the Unconstrained Unit Cell refinement.

1. View the text output:
   - At the bottom of the SaintChart window is the scrolling text window that displays text output (this window can be expanded by dragging the horizontal bar above the text).
   - Alternatively, double-click any of the output entries (in the “Output Filename” area) for an additional tool: the **ls** button at the far right of the line. The **ls** button opens the listing file for that integration run.

Figure 6.14 —Runs List’s **ls** button

6.7.1 Overall R_{sym} and Coverage Statistics

The overall R_{sym} is usually less than 8%. Higher values may indicate problems with absorption, twinning, or poor crystal quality.

The coverage statistics are reported by resolution shells:

- The values in the `Rsym` column give the cumulative agreement of equivalent reflections.
- The `Rshell` values give agreement within a particular shell. These change faster than the `Rsym` values.
- The `%<2s` column is useful for checking where the weak data becomes predominant (greater than 50 or 60%).
- The `#Sigma` column gives the average I/sigma for each shell. If that number is less than 1.5 or 2, then little significant data is being measured in that shell.

Figure 6.15 —Final SAINT Overall R_{sym} and coverage statistics

```
Integration of ylid
 ...1.1(1):  component 1 in sample 1 (component 1 in ylid_0m.raw)
   Overall      #  Pairs   Uniq   Merg %<2s       <I> <#Sig>    <Bg>  Rsym   dI/I dI/s    R+ Ranom Canom ErX ErY ErZ RmX RmY RmZ
     0.000   8142   1363   1436   8100  7.0  4797.709  42.21    2.60 0.022 -0.000  0.7 0.020 0.026  0.95-.00-.00 .00 .21 .17 .11
     1.000

Integration of ylid
 ...1.1(1):  component 1 in sample 1 (component 1 in ylid_0m.raw)
   Centric      #  Pairs   Uniq   Merg %<2s       <I> <#Sig>    <Bg>  Rsym   dI/I dI/s    R+ Ranom Canom ErX ErY ErZ RmX RmY RmZ
     0.000   1598    392    442   1575 14.6  7230.902  61.01    2.65 0.019 -0.000  0.8 0.015 0.028  1.21-.01-.01 .00 .25 .20 .14
     1.000

Coverage Statistics
Integration of ylid
 ...1.1(1):  component 1 in sample 1 (component 1 in ylid_0m.raw)
                                                                  .....Shell.....
  Angstrms   #Obs Theory %Compl Redund   Rsym Pairs %Pairs Rshell #Sigma %<2s
to  1.618    179    179 100.00   7.35  0.017   177  98.88  0.017 107.77  3.7
to  1.284    329    329 100.00   7.35  0.018   325  98.78  0.022  43.11  2.4
to  1.122    476    476 100.00   7.12  0.019   471  98.95  0.024  29.99  2.2
to  1.019    619    619 100.00   6.90  0.020   613  99.03  0.030  20.15  5.4
to  0.946    769    769 100.00   6.62  0.020   760  98.83  0.039  16.01  7.2
to  0.890    910    910 100.00   6.43  0.021   896  98.46  0.043  12.00  8.5
to  0.846   1043   1043 100.00   6.24  0.021  1023  98.08  0.058   9.17  8.2
to  0.809   1195   1195 100.00   6.05  0.022  1164  97.41  0.062   8.12 12.6
to  0.778   1327   1327 100.00   5.89  0.022  1288  97.06  0.068   6.68 18.6
to  0.751   1436   1462  98.22   5.67  0.022  1363  93.23  0.082   5.78 15.3
```

6.7.2 Unconstrained Unit Cell Refinement

Check the angles in the unconstrained unit cell refinement to get a quick confirmation that the initial lattice determination was correct. In the unconstrained refinement, angles that are required to be 90° or 120° are allowed to refine. If they differ considerably from the expected value, then the data should be checked carefully.

Figure 6.16 —Unconstrained unit cell refinement

```
Unconstrained global unit cell refinement ============== 04/07/10 09:49:48

Performing final unit cell least squares on file C:\Frames\guest\ylid\work\ylid_01._ma
Input file contains       4814 reflections

Refinement includes 1 sample
   Sample 1 contains component   1
Maximum allowed reflections =       9999
One reflection will be stored in memory per     1 reflection(s) read

.................
Global Refinement, sample 1 of 1 -- single-component data
This sample is a single crystal with 3 indices (HKL)
Orientation LS for Sample 1 of 1, component 1 in sample, 1 in .p4p/.spin file (4814 input reflections)...

Relative SVD-filter threshold:     0.0000010
Starting gradient-expansion term:  0.0020000
Weighting multipier for Z terms:   1.0000000
Minimum elements to filter:                0
Maximum number of LS cycles:              25
Vertical tilt of beam (deg):          0.0000
Individual reflection weights will be used in the refinement

Wavelength, relative uncertainty:  0.7107300,  0.0000089

Component numbers S.C(F) below: S=sample, C=component in sample, F=component in file

Reflection Summary:
'RLV.Excl' are reflections excluded after cycle 1 because RLV error exceeded 0.0250:
 Component      Input  RLV.Excl      Used  WorstRes   BestRes   Min.2Th   Max.2Th
    1.1(1)       4814         0      4814    9.1952    0.7558     4.430    56.096

Orientation ('UB') matrix:
  -0.0005221   0.0652497  -0.0439144
   0.1290755   0.0572461   0.0203791
   0.1071686  -0.0686226  -0.0247590

         A          B          C      Alpha       Beta      Gamma        Vol
    5.9606     9.0374    18.3903    90.0024    89.9999    90.0025     990.66
    0.0004     0.0006     0.0012     0.0007     0.0007     0.0008       0.20
Corrected for goodness of fit:
    0.0002     0.0003     0.0006     0.0004     0.0004     0.0004       0.10

Crystal system constraint:            0  (Unconstrained)
Parameter constraint mask:            0  (0 = 0[hex])

Eulerian angles (deg):           65.108     117.084      45.846
Goniometer zeros (deg):          0.0000*     -0.0123      0.0000*    -0.0002    (*=never refined)
Crystal translations (pixels):               -0.0542     -0.0370      0.0387

Detector corrections:   X-Cen    Y-Cen     Dist    Pitch     Roll      Yaw
                     -0.2232   0.0319  -0.0257   0.0031   0.1555   0.1870

Refinement statistics:   StartingRes      FinalRes     GOF #Cycles #Filtered    Code
                        3.53716e+003  3.51135e+003    0.50       3         0       0

Overall statistics for terms in X, Y, and Z:       X             Y             Z
 Number of nonzero-weight terms:              4814          4814          4649
 Average input ESD (pix, pix, deg):        0.10564       0.10239       0.03753
 Goodness of fit:                          0.56732       0.52470       0.37005

Highest-magnitude correlations:
  1 -0.98851 Distance1 / X-Center1
  2 -0.98763 X-Center1 / C1
  3 -0.98665 X-Center1 / B1
  4  0.98636 Distance1 / C1
  5  0.98586 Distance1 / B1
  6 -0.98570 X-Center1 / A1
  7  0.98422 Distance1 / A1
  8  0.98265 C1 / B1
  9  0.98155 B1 / A1
 10  0.98108 C1 / A1

New orientation is in C:\Frames\guest\ylid\work\ylid_0u.p4p
```

Unit cell parameters

6.8 Scaling Data

The scaling process puts all of the measured data on the same scale. The Scale module exploits data redundancy to correct 3D-integrated data and provide useful diagnostics such as:

- Variations in the crystal's volume;
- Incident beam inhomogeneities;
- Absorption by the crystal support; and
- Crystal decay.

The only required inputs for the Scale module are a .raw file (output from the Integrate Images module) and the crystal's Laue group. For a more detailed description of the module's parameters, refer to the SADABS documentation file provided.

Scaling involves five steps:

1. Setting up input files
2. Refining parameters
3. Refining the error model
4. Displaying diagnostics
5. Exiting

6.8.1 Start the Scale Module

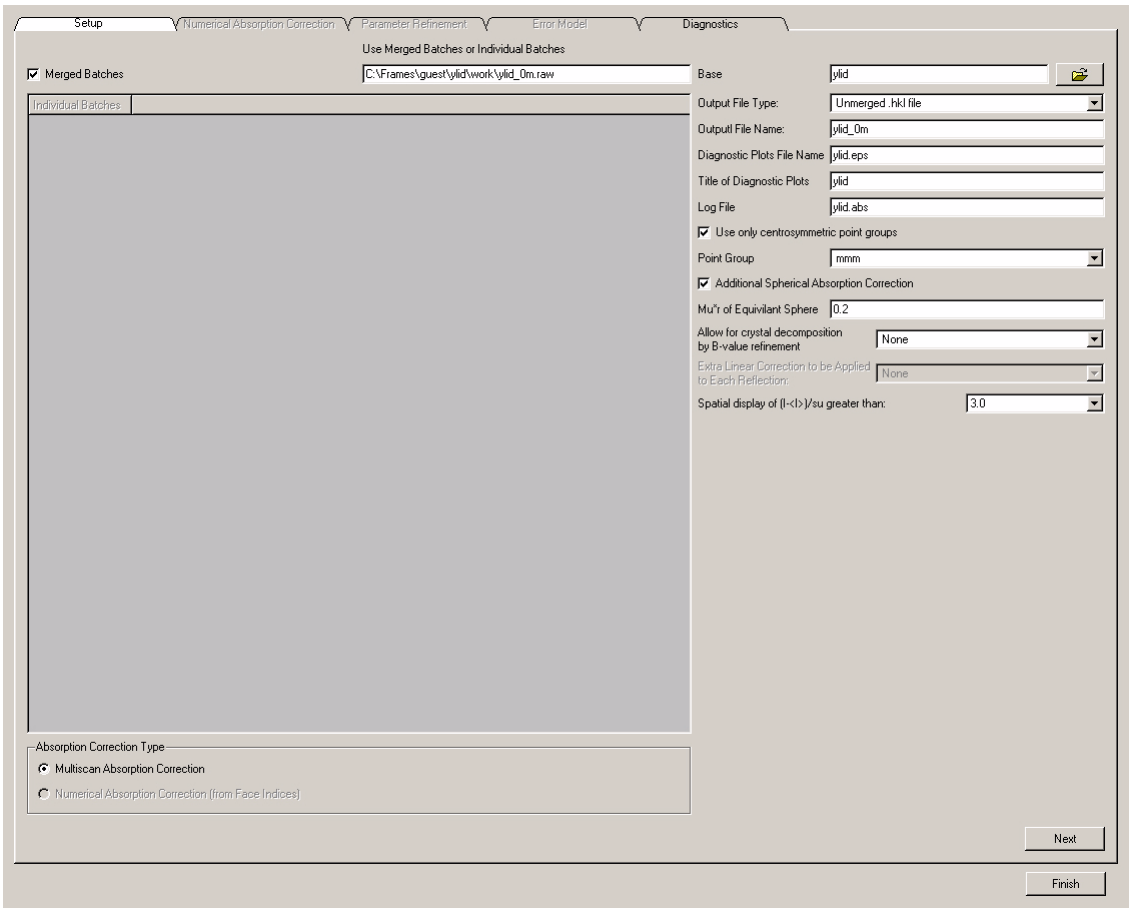
1. In the Task Bar's "Scale" category, click the **Scale** icon .

   The Scale module opens.

6.8.2 Set Up the Scale Operation

Figure 6.17 shows the right-hand portion of the module's "Setup" tab.

Figure 6.17 —"Setup" tab



1. Check that the defaults are correct.

   Generally, the Absorption Correction Type is the only value that might be changed. Numerical Absorption Correction requires the Crystal Faces module.

2. If necessary, read in the runs to be scaled:

   Click the “Base:” field’s **Browse** button . A window appears showing the .raw files available for scaling. If the files are not visible, select the correct folder. A single run can be selected by highlighting that run and clicking **OK**. All runs with the same base name can be chosen by highlighting any of the runs in that series and activating the **Open All With Base** checkbox. Selecting a merged file (i.e., the one ending in “m” rather than a run number) will use all of the runs that were integrated during the same integration job. The program will assign filenames for the output files. To change these names, edit the output boxes.

3. Choose the preferred point group. To keep Friedel pairs separate during scaling, deactivate the **Use only centrosymmetric point groups** checkbox.

4. If necessary, activate the **Additional Spherical Absorption Correction** checkbox.

   It is possible to apply an additional absorption correction assuming a spherical crystal with given μ*r*, where μ is the linear absorption coefficient and *r* is the radius of the equivalent sphere. This correction is included because the theta-dependent part of the absorption cannot be modeled well by comparing equivalent reflections (because these invariably have the same 2theta values). The main effect of applying it will be to increase the equivalent isotropic displacement parameters in the resulting refinement.

NOTE: This parameter should be disabled unless the sample contains a strong absorber. A good starting value for the “Mu*r of Equivalent Sphere” field is `0.05`. Check to see if the min/max transmission ratio is closer to 1.0.

5. If necessary, set the **Allow for crystal decomposition by B-value Refinement** parameter.

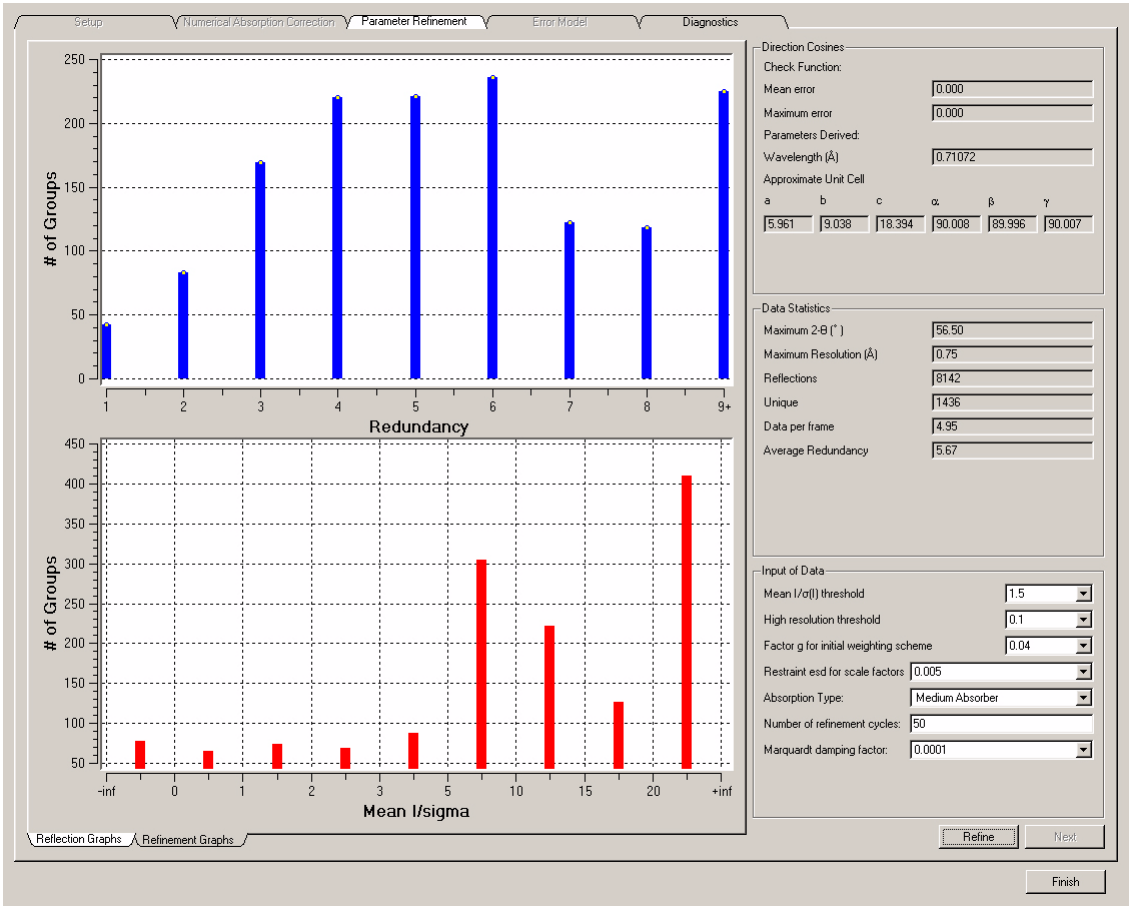
6. Click **Next**.

   The module proceeds to the “Parameter Refinement” tab.

6.8.3 Refine the Parameters

The “Parameter Refinement” tab shows the reflection statistics before refinement, under the “Reflection Graphs” sub-tab.

Figure 6.18 —“Parameter refinement” tab before refinement



1. If necessary, set the refinement parameters (the defaults work well in most cases):

Table 6.3 — Refinement parameters

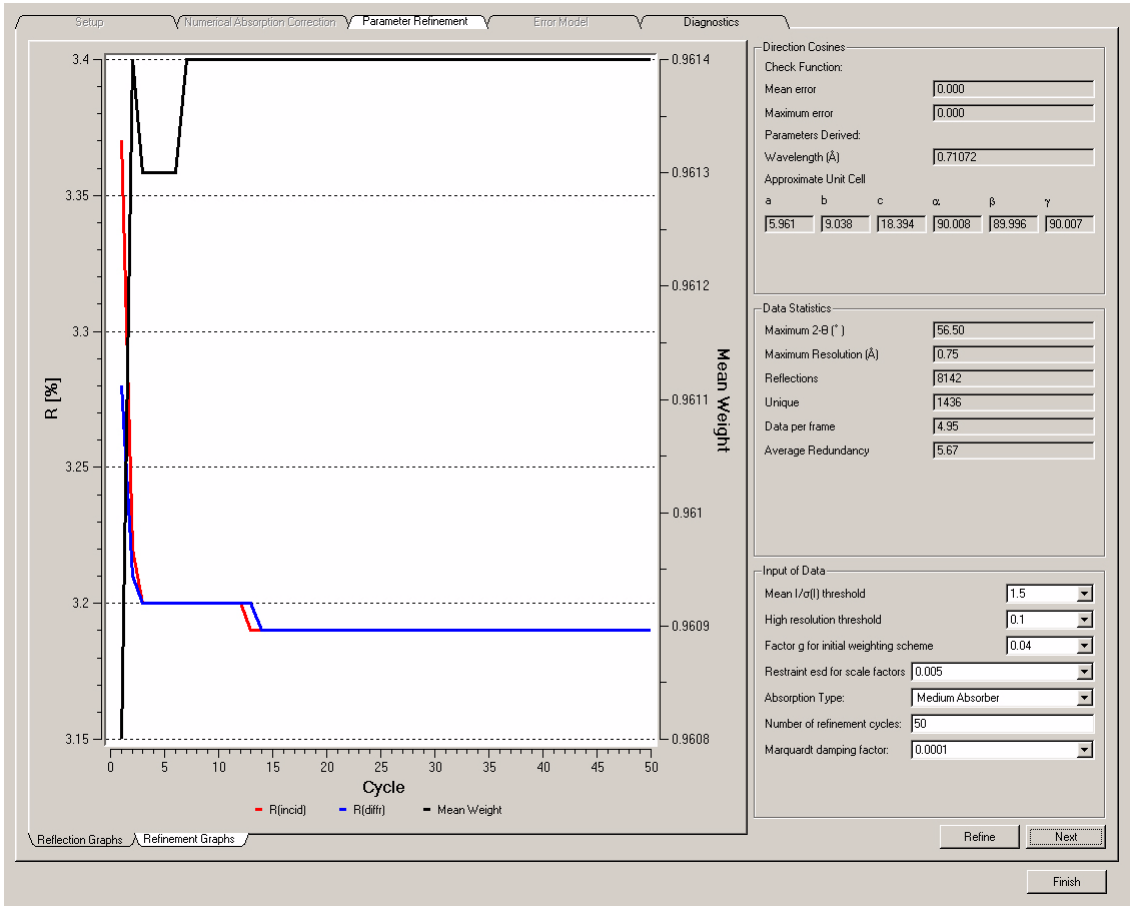
Parameter	Usage
Mean I/sigma(I) threshold	The signal/noise cutoff for reflections used in the scale factor determination.
High Resolution threshold	The resolution cutoff for reflections used in the scale factor determination.
Factor g for initial weighting scheme:	Read the SADABS documentation for how this weighting factor is used.
Restraints esd for Scale Factors	Restrains the scale factors from shifting too much during refinement. Relaxing the restraints a bit may help in some cases, but be careful not to over-fit the data.
Absorption Type	Sets the order of spherical harmonics used for the absorption correction. Medium is fine for all data sets. If there are heavy atoms in the unit cell and the data redundancy is high, the Strong Absorber setting may help.
No. of Refinement Cycles	Sets the number of cycles of scale factor refinement.
Marquardt damping factor	Damping parameter for the Levenberg-Marquardt algorithm used in least-squares analysis. The damping factor is adjusted so that the closer you get to optimum, the more damping takes place.

2. Click **Refine**.

   The module proceeds to the “Refinement Graphs” tab.

   Figure 6.19 shows a typical refinement result. The R-values should decrease quickly and converge to similar values. The Mean Weight should increase and converge. Typical final values are 3–4% for the R-values, and 0.95–0.98 for the Mean Weight.

Figure 6.19 —“Refinement Graphs” tab after refinement



3. If necessary, make changes to the refinement parameters and click **Refine** to refine the data set again.

4. When you are satisfied with the refinement, click **Next**.

   The module proceeds to the “Error Model” tab.

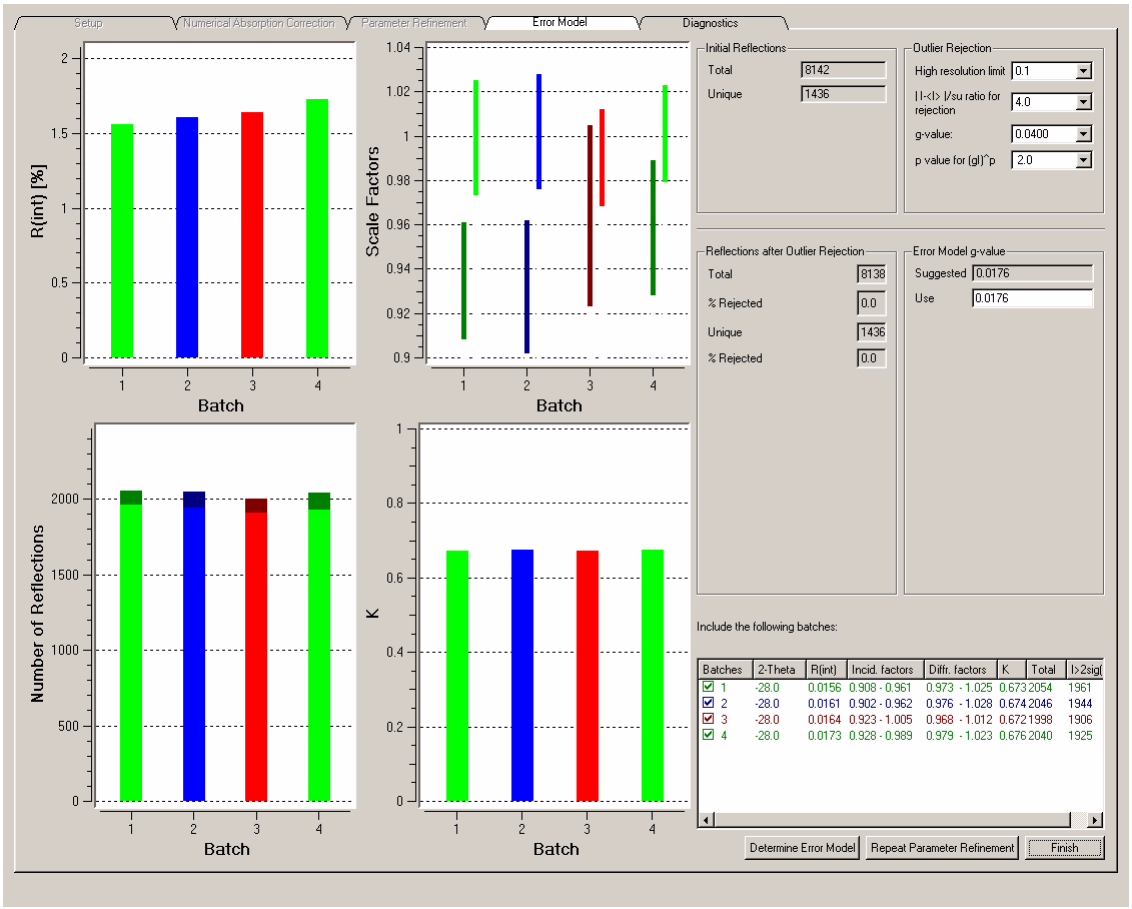
6.8.4 Refine the Error Model

Initially, the graphs in the “Error Model” area are blank.

1. If necessary, adjust the parameters in the “Outlier Rejection” area.
2. Click **Determine Error Model**.

   Figure 6.20 shows typical Error Model results.

Figure 6.20 —Error Model results



3. If necessary, adjust the error model and click **Determine Error Model** again.

   For example, to reject fewer reflections, increase the |I-<I>|/su ratio in the “Outlier Rejection” area.

NOTE: If the R-values in one run are significantly higher than the others, you can repeat the entire scaling calculation with that run omitted. Deactivate the checkbox next to the “bad” run, and click **Repeat Parameter Refinement**.

4. When you are satisfied with the error model, click **Finish**.

   The module proceeds to the “Diagnostics” tab.

6.8.5 Examine the Diagnostic Plots

Diagnostic plots provide valuable insight into the quality of the data and possible problems with the data.

1. Click the tabs at the bottom of the screen to view the diagnostic data (some of these plots are reviews of plots that have already been displayed).

Scale Variations

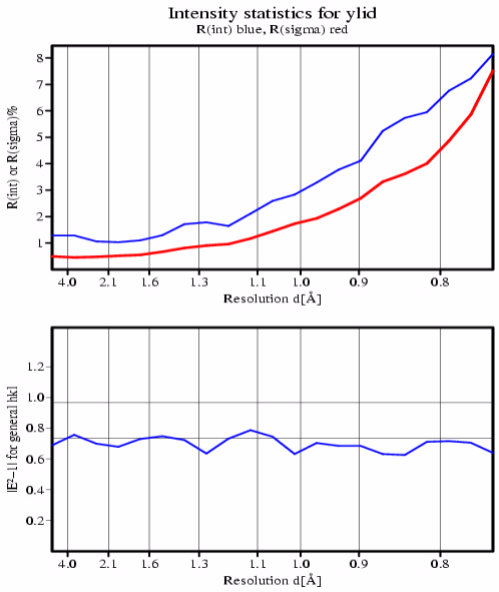
The Scale Variations plot (Figure 6.21) shows the overall variation in Scale and R(int) for the individual frames. The Scale plot should be flat (for strongly-absorbing, irregularly-shaped crystals it will probably be sinusoidal), and the R(int) plot should not show large variations (more than 2%).

Figure 6.21 —Overall Scale and R(int) variations

Intensity Statistics

The Intensity Statistics plots look at R(int) and $|E^2-1|$ as a function of resolution. Typical plots of R(int) versus resolution increase to the right as seen in Figure 7.24. An $|E^2-1|$ plot should have a constant value. $|E^2-1|$ is a strong indicator for centric and acentric space groups. The two horizontal lines indicate the expected values for centric (top) and acentric (bottom) space groups. This plot clearly suggests an acentric space group.

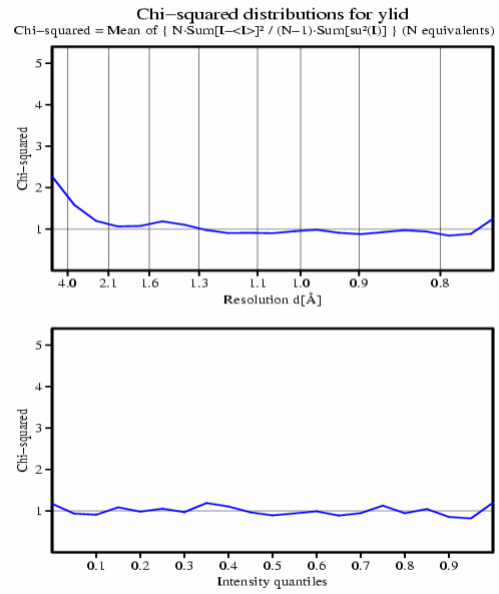
Figure 6.22 —Intensity Statistics



Chi-Squared Distributions

The plots of Chi-Squared values for the data as a function of resolution and intensity should be mostly flat. The plots shown in Figure 6.23 are typical.

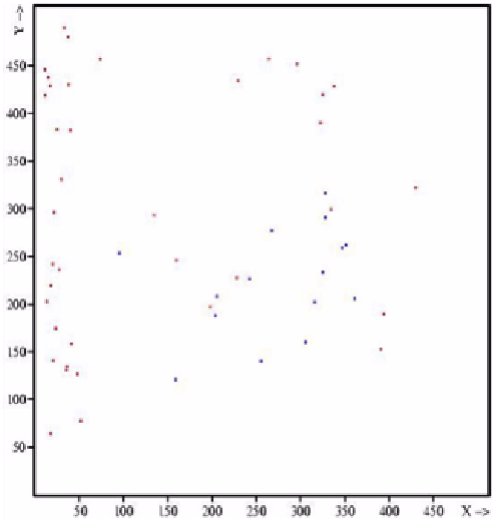
Figure 6.23 —Chi-squared distributions



Spatial Distribution Plots

Spatial Distribution plots are generated for each data collection run. They indicate spots that were either stronger or weaker than expected with a deviation from the mean intensity larger than three standard uncertainties.

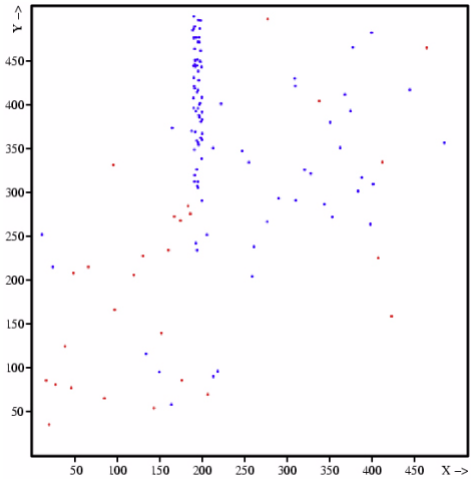
Figure 6.24 —Spatial Distribution plots



Typically, the points of disagreement are spread fairly evenly over the detector face.

If the points are clustered in a single area or if there are significantly more spots of one color than another, the data should be examined critically. Figure 6.25 is from a data set in which the Active Mask was not used during integration. Consequently, the reflections collected in that area are consistently weaker than expected. This kind of problem may affect the final results.

Figure 6.25 —Spatial Distribution plot showing a problem area



6.8.6 Close the Scale Module

1. Click **Exit AXScale**.

   The module closes.

This page intentionally left blank.

7 Examining Data

After integrating and scaling the data, the crystal's space group is determined and optional simulated precession photographs are calculated to further evaluate the overall quality of the data. Three modules are available for this:

- Space Groups and Statistics (based on XPREP);
- Space Group Determination (which opens the command-line version of XPREP); and
- Precession Images (for looking at undistorted slices of reciprocal space).

7.1 Using the Space Groups and Statistics Module

7.1.1 Start the Space Groups and Statistics Module

1. In the Task Bar, click the **Space Groups and Statistics** icon .

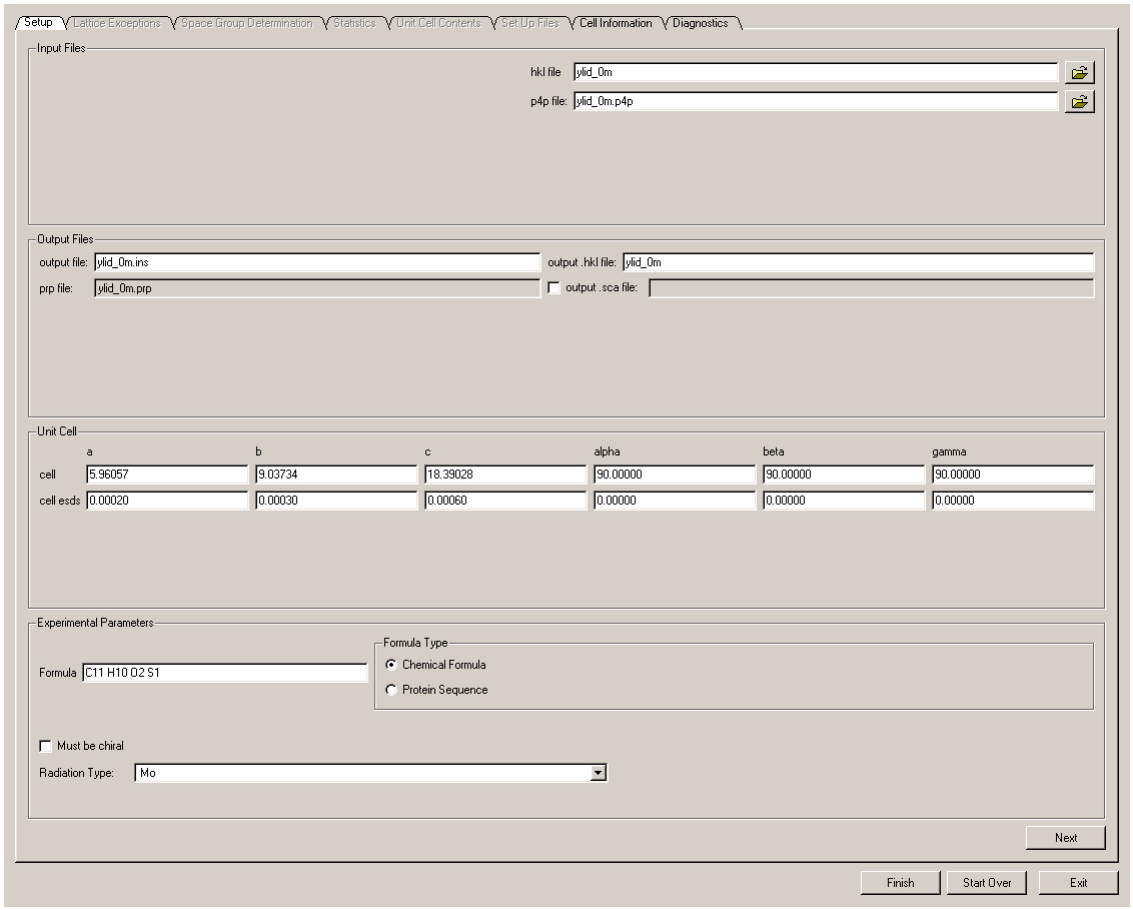
   The module opens with the “Setup” tab active.

7.1.2 Set Up the Determination Parameters

1. In the module’s “Setup” tab, check that the default-loaded files are correct.

NOTE: Typically the files to use for space group determination are the “/work” directory’s *m.p4p file (containing the final unit cell parameters from integration) and *m.hkl file (containing the corrected intensities). You can browse to choose other files if desired.

Figure 7.1 — “Setup” tab



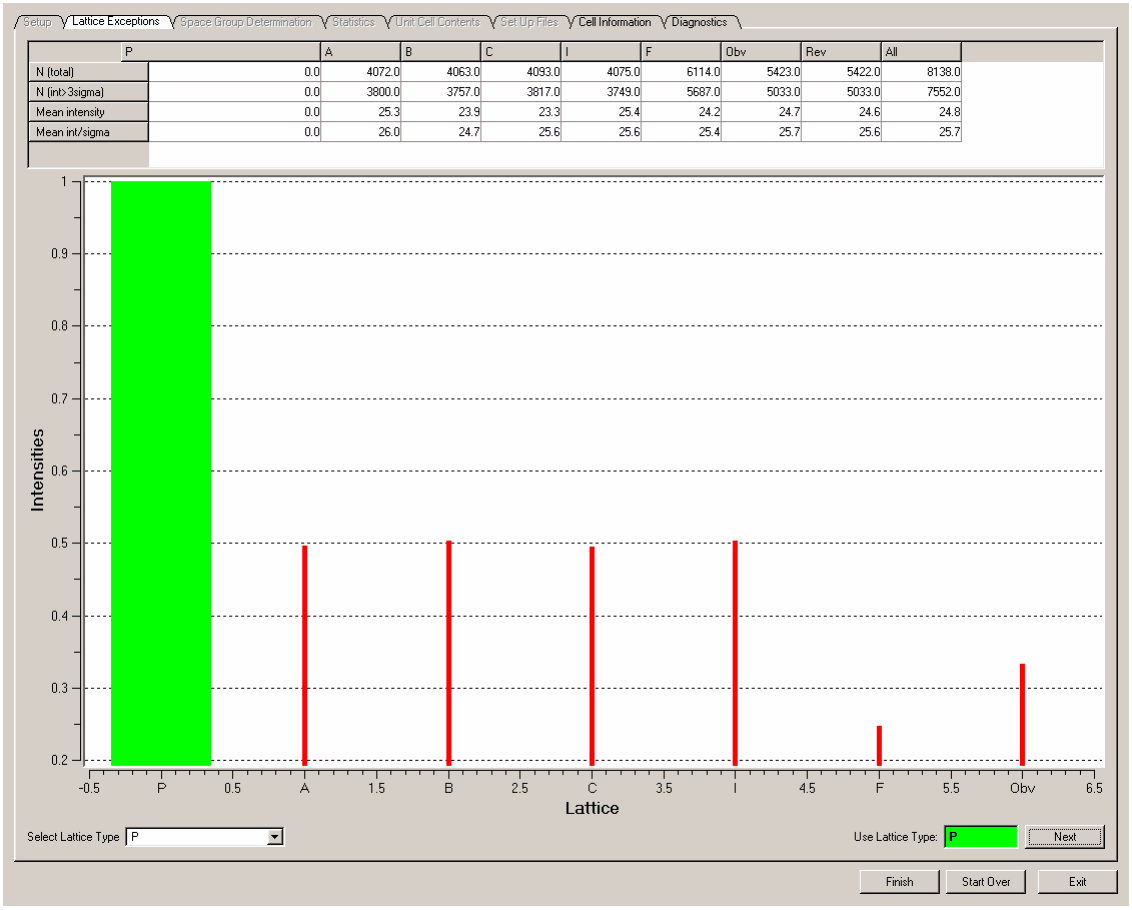
2. Click **Next**.

   The module proceeds to the “Lattice Exceptions” tab.

7.1.3 Examine the Lattice Exceptions

The module evaluates the data and suggests a lattice type based on the mean intensities and the mean int/sigma.

Figure 7.2 — “Lattice Exceptions” tab



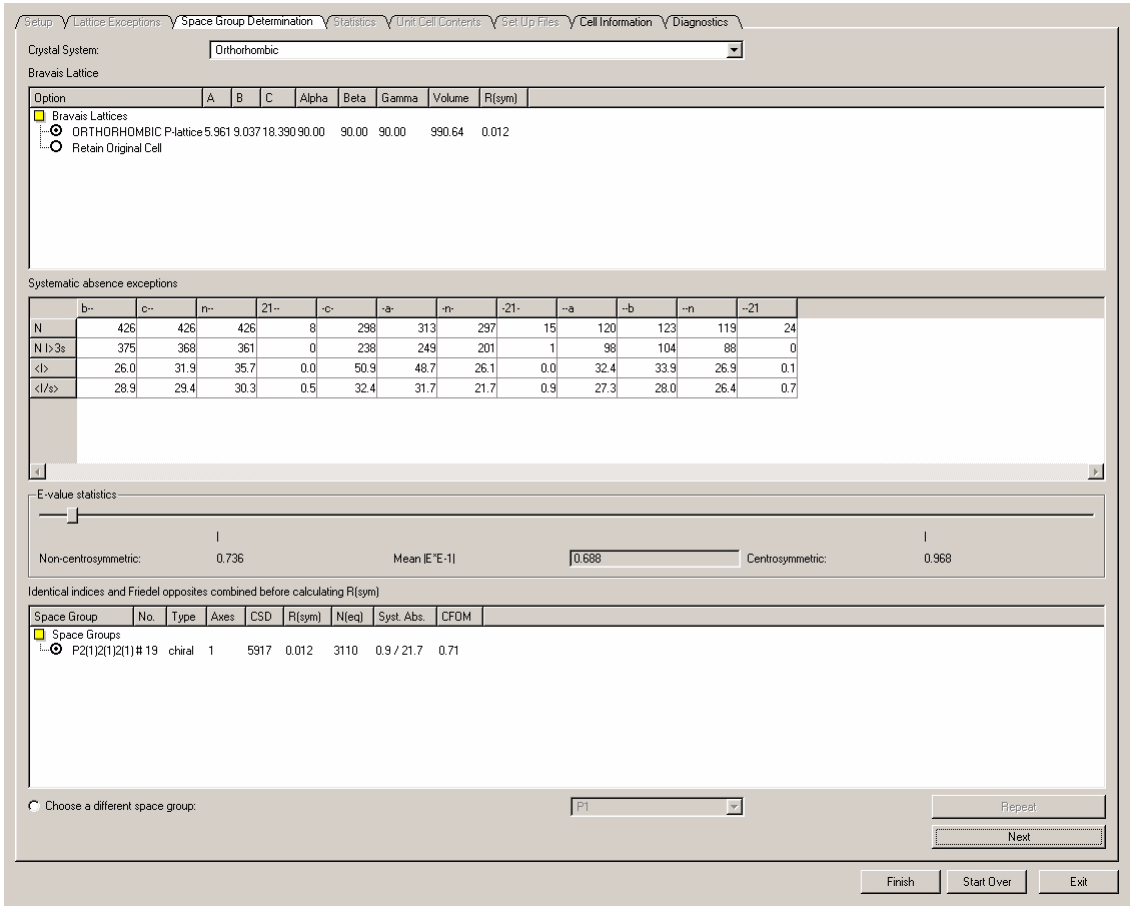
1. If you know that your sample has a different lattice type than that suggested by the module, you can choose it from the **Select Lattice Type** pull-down menu.
2. Click **Next**.

   The module proceeds to the “Space Group Determination” tab.

7.1.4 Choose a Space Group

The module determines the reduced unit cell based on the chosen lattice type.

Figure 7.3 — Space group determination



1. Check the E-value statistics to determine if the space group is centrosymmetric or non-centrosymmetric.
2. At the bottom of the Task Display Area, examine the suggested space groups and choose a space group by clicking its radio button.

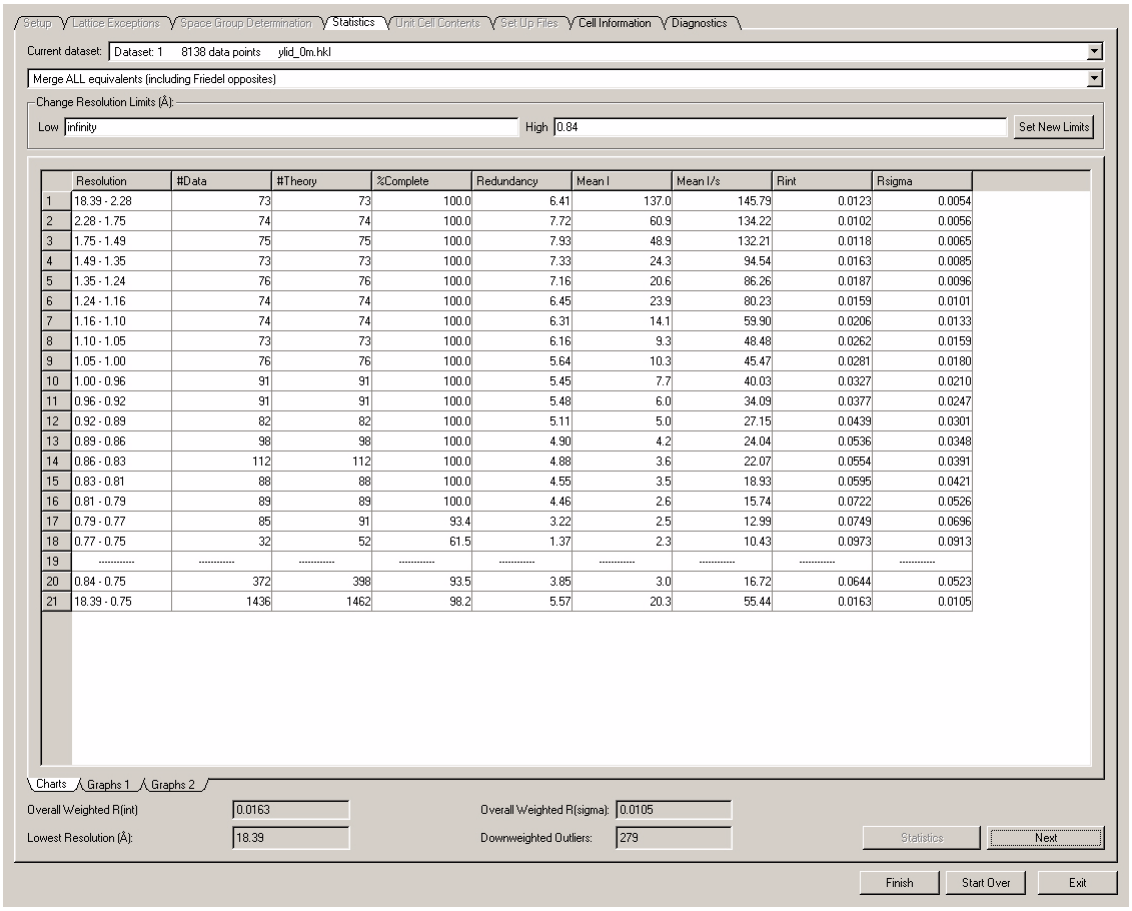
NOTE: To choose a space group other than the suggested ones, click the **Choose a different space group** radio button. This opens a drop-down menu of all permissible space groups.

3. Click **Next**.

   The module proceeds to the “Statistics” tab.

7.1.5 Examine the Reflection Statistics

Figure 7.4 — "Statistics" tab



	Resolution	#Data	#Theory	%Complete	Redundancy	Mean I	Mean I/s	Rint	Rsigma
1	18.39 - 2.28	73	73	100.0	6.41	137.0	145.79	0.0123	0.0054
2	2.28 - 1.75	74	74	100.0	7.72	60.9	134.22	0.0102	0.0056
3	1.75 - 1.49	75	75	100.0	7.93	48.9	132.21	0.0118	0.0065
4	1.49 - 1.35	73	73	100.0	7.33	24.3	94.54	0.0163	0.0085
5	1.35 - 1.24	76	76	100.0	7.16	20.6	86.26	0.0187	0.0096
6	1.24 - 1.16	74	74	100.0	6.45	23.9	80.23	0.0159	0.0101
7	1.16 - 1.10	74	74	100.0	6.31	14.1	59.90	0.0206	0.0133
8	1.10 - 1.05	73	73	100.0	6.16	9.3	48.48	0.0262	0.0159
9	1.05 - 1.00	76	76	100.0	5.64	10.3	45.47	0.0281	0.0180
10	1.00 - 0.96	91	91	100.0	5.45	7.7	40.03	0.0327	0.0210
11	0.96 - 0.92	91	91	100.0	5.48	6.0	34.09	0.0377	0.0247
12	0.92 - 0.89	82	82	100.0	5.11	5.0	27.15	0.0439	0.0301
13	0.89 - 0.86	98	98	100.0	4.90	4.2	24.04	0.0536	0.0348
14	0.86 - 0.83	112	112	100.0	4.88	3.6	22.07	0.0554	0.0391
15	0.83 - 0.81	88	88	100.0	4.55	3.5	18.93	0.0595	0.0421
16	0.81 - 0.79	89	89	100.0	4.46	2.6	15.74	0.0722	0.0526
17	0.79 - 0.77	85	91	93.4	3.22	2.5	12.99	0.0749	0.0696
18	0.77 - 0.75	32	52	61.5	1.37	2.3	10.43	0.0973	0.0913
19	.....	.....	.....	.....	.....	.....	.....	.....	.....
20	0.84 - 0.75	372	398	93.5	3.85	3.0	16.72	0.0644	0.0523
21	18.39 - 0.75	1436	1462	98.2	5.57	20.3	55.44	0.0163	0.0105

1. Adjust the statistics parameters if desired:
   - The two pull-down menus at the top of the "Statistics" tab allow you to change the current dataset and control merging of equivalent reflections.
   - Change Resolution Limits allows you to adjust the range of resolutions for which statistics are displayed. Adjust the quantities in the fields and click **Set New Limits**.
2. Examine the data presented in the table of statistics and in the "Graphs" tabs along the bottom of the screen:
   - Is the completion near 100%?
   - Are the redundancies at various resolutions close to the desired values?
   - Are R_{int} and R_{sigma} small and increasing smoothly from top to bottom (low-angle to high-angle data)?
   - Are the Overall Weighted R(int), Overall Weighted R(sigma), Lowest Resolution (Å) and Down-weighted Outliers appropriate for your experiment?

Figure 7.5 — Graphs 1

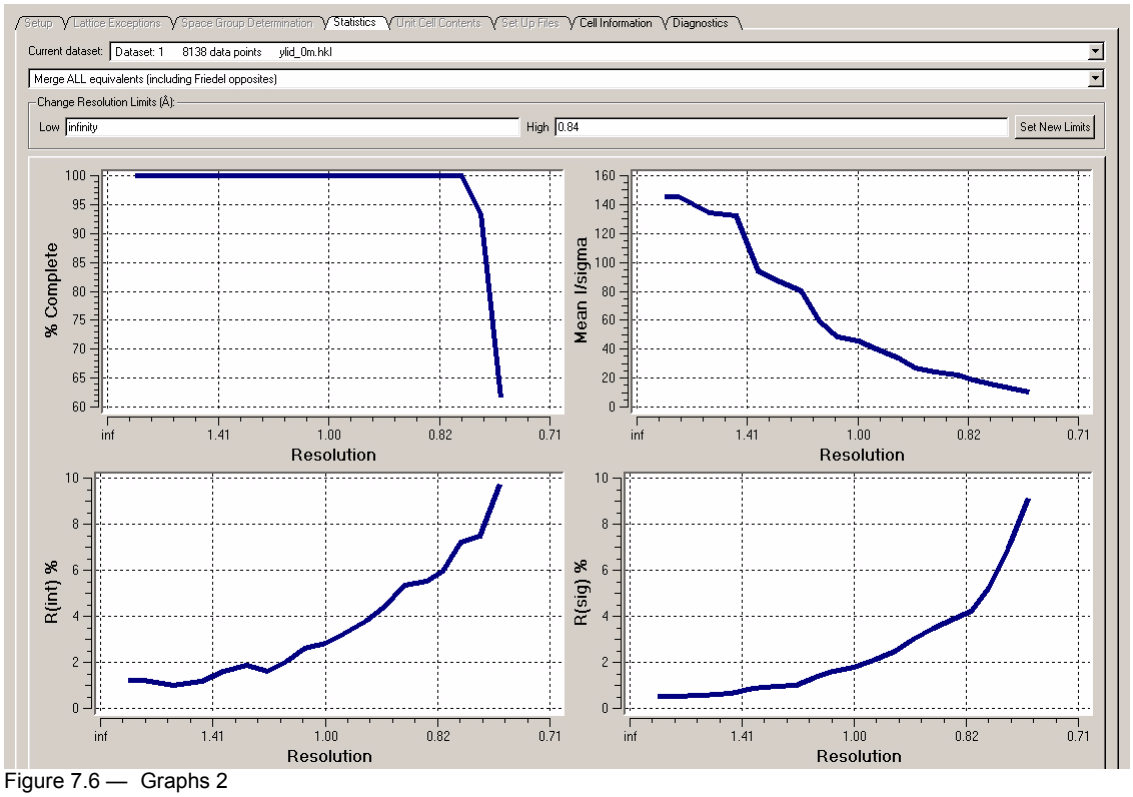
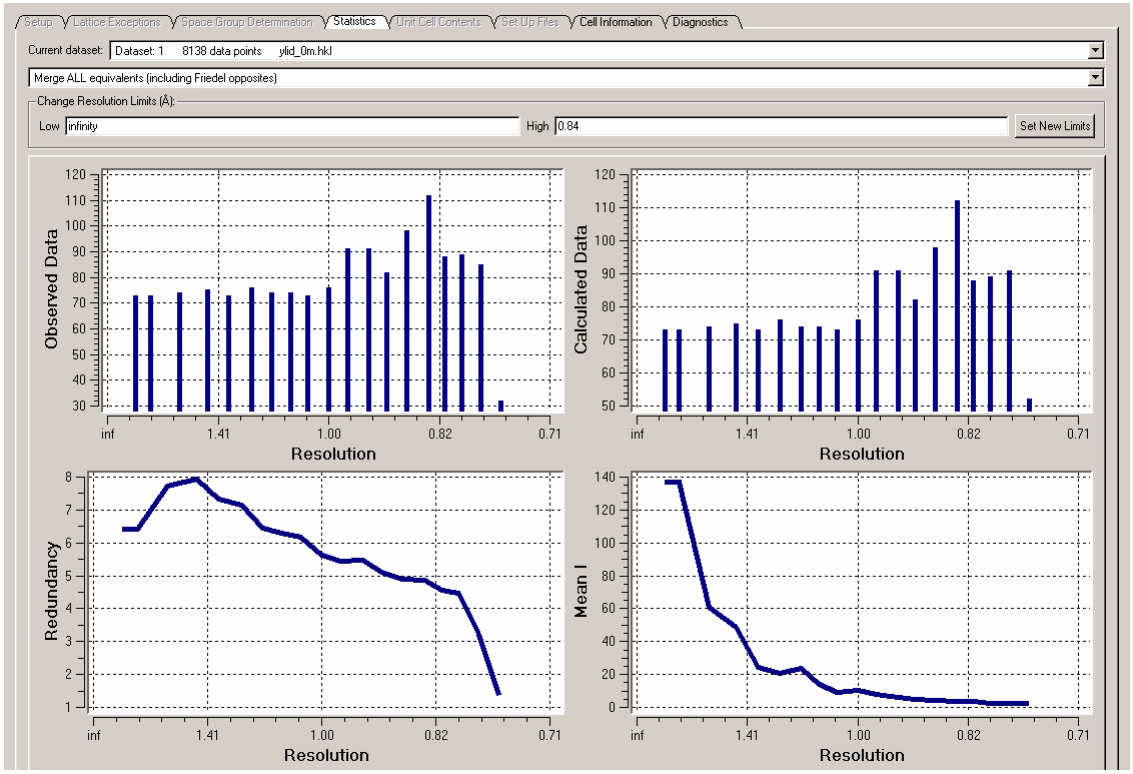


Figure 7.6 — Graphs 2



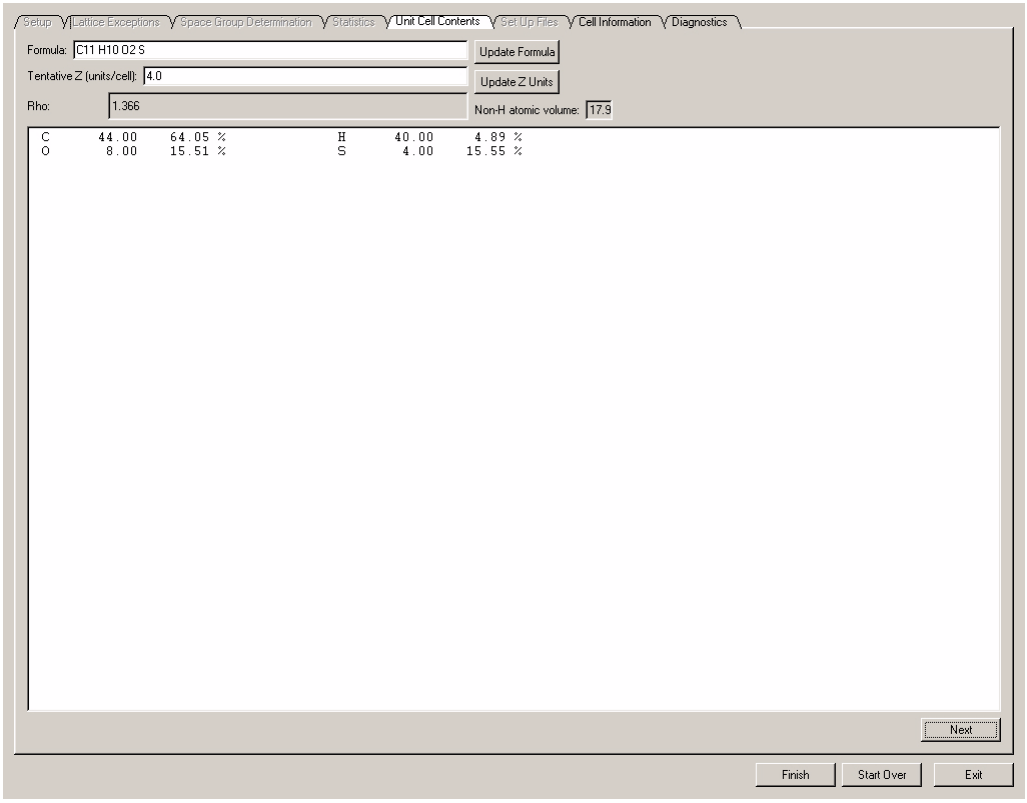
3. Click the **Next** button.

   The module proceeds to the “Unit Cell Contents” tab.

7.1.6 Check the Unit Cell Contents

The “Unit Cell Contents” tab displays the current formula from the module’s “Setup” tab, a tentative Z value, the density, and the atomic volume.

Figure 7.7 — “Unit Cell Contents” tab



1. Check that the information is correct:

   1.1. Check that Z seems reasonable for the space group, that the Rho value (density) is as expected (1.1 to 1.4 for organic molecules, higher for inorganic compounds).

   1.2. Check that the Non-H atomic volume is around 17 or 18.

   Significant variation from the expected values may indicate an incorrect molecular formula.

2. If necessary, update the formula or Z units by typing directly in the field(s) and clicking the appropriate buttons at the top of the module.

3. Click **Next**.

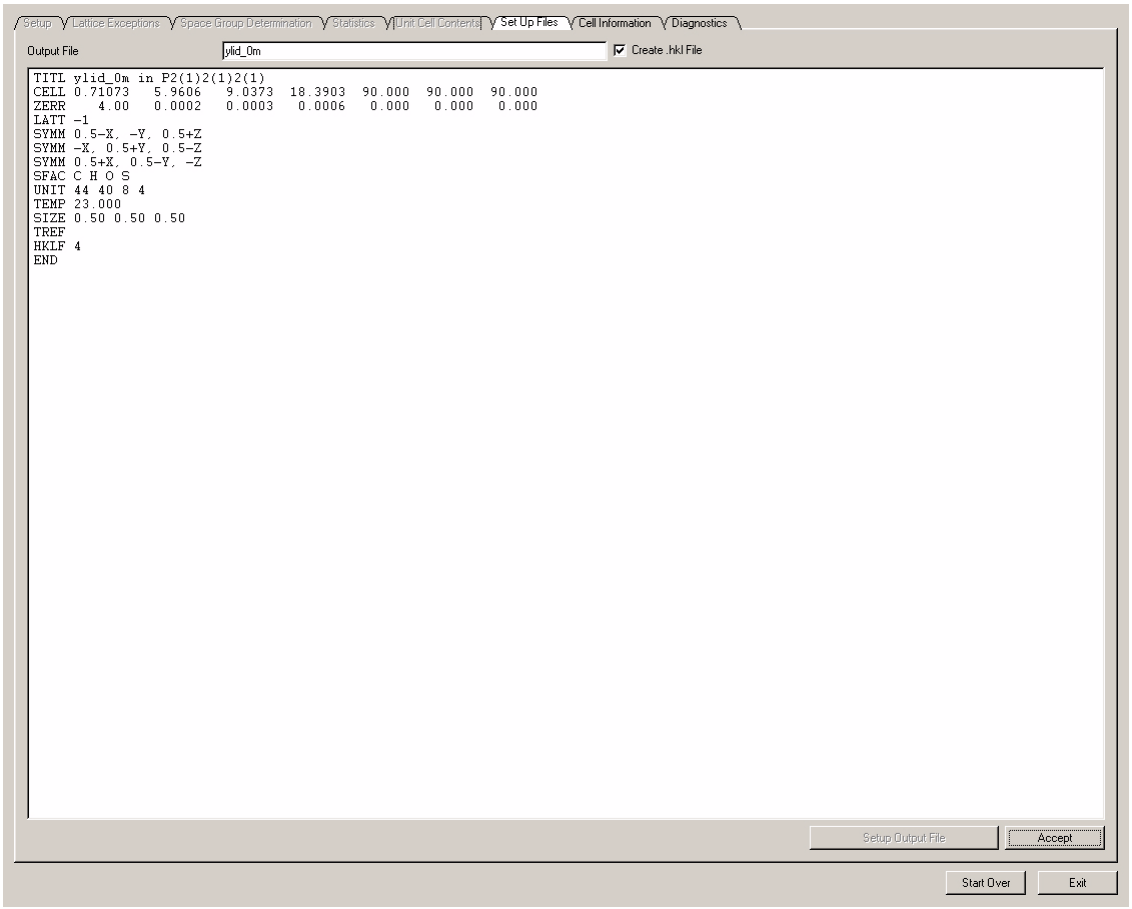
   The module proceeds to the “Set Up Files” tab.

7.1.7 Set Up the Output File

1. If you want to change the output file name, type the new name in the “Output File” field.
2. If you want to overwrite the existing .hkl file, activate the **Overwrite Existing .hkl File** checkbox.
3. Click **Setup Output File**.

   The module fills in the appropriate commands for the .ins file.

Figure 7.8 — “Set Up Files” tab (filled in)

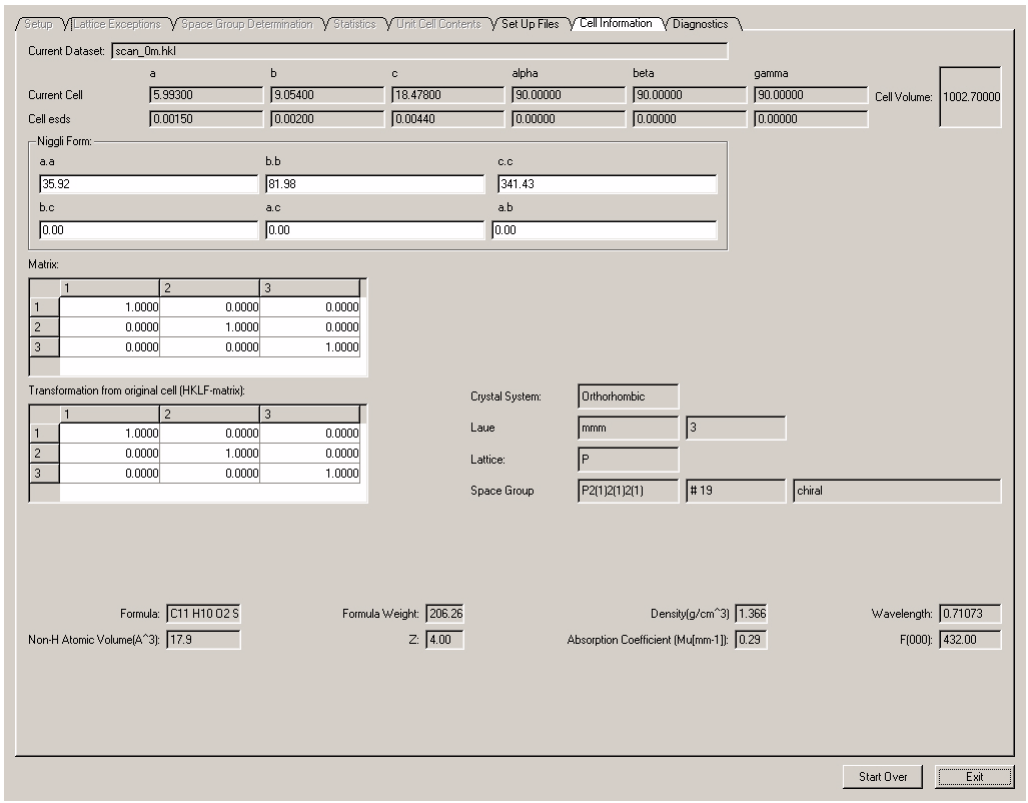


4. Click **Accept** to write the .ins file.

7.1.8 Examine Cell Information

1. Click the “Cell Information” tab to examine additional cell information.

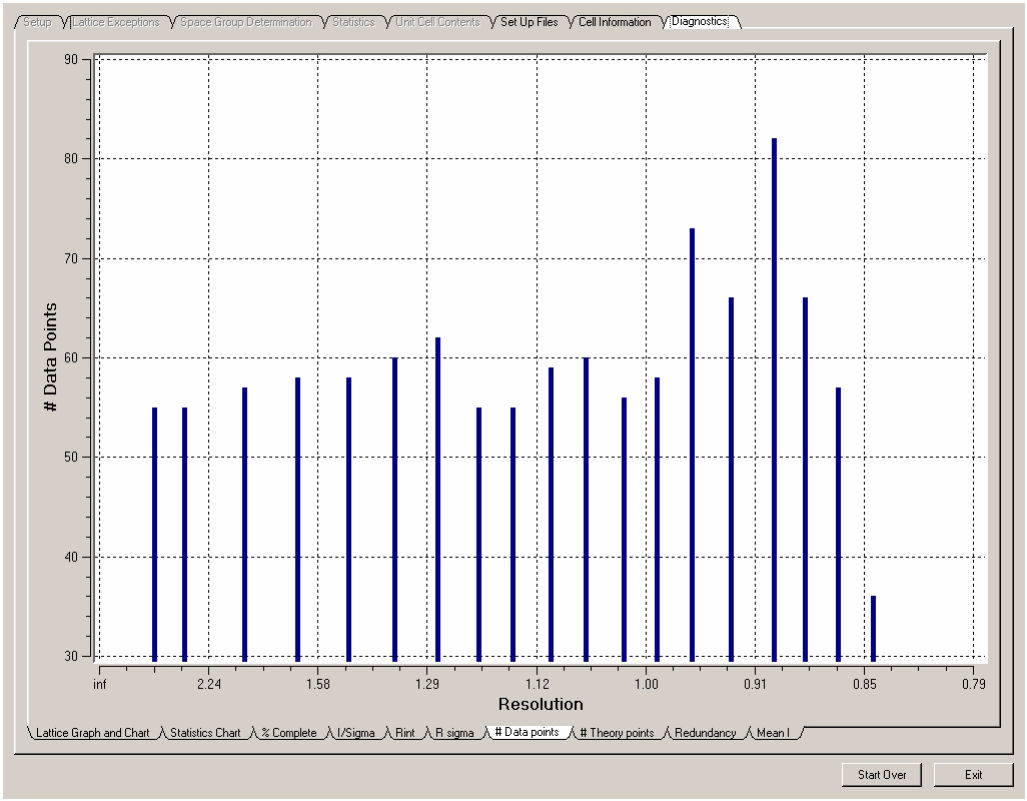
Figure 7.9 — “Cell Information” tab



7.1.9 Examine Diagnostics

1. Click the “Diagnostics” tab to examine additional diagnostic information.

Figure 7.10 —“Diagnostics” tab (“# Data Points” Section shown)



7.1.10 Exit the Space Groups and Statistics Module

1. Click the **Exit** button in the module’s lower right-hand corner.

   The module closes.

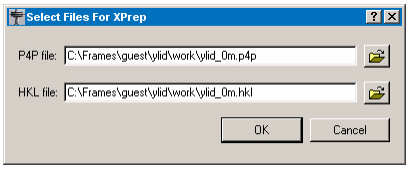
7.2 Determining the Space Group with XPREP

1. Under the Task Bar's "Examine Data" category, click the **Space Group Determination** icon .

   The module starts, and the "Select Files for XPrep" window opens.

2. Check that the two files in the "Select Files for XPrep" window are correct for your data set (Figure 7.11) and click **OK**.

Figure 7.11 — "Select Files for XPrep" window



NOTE: In this example, the integration process has created two files: ylid_0m.p4p containing the final unit cell parameters from integration and ylid_0m.hkl containing the corrected intensities. Typically, these are the files to use for determining space groups, but you can browse to choose other files.

NOTE: In addition to space group determination, many other features of XPREP can be accessed from the general menu. Resolution cutoff, reciprocal space plots, simulated powder patterns, and a test for merohedral twinning are very useful tools.

7.2.1 Determine the Space Group

1. Lattice type: XPREP evaluates the data and looks at the mean intensities and the mean int/sigma. Since these are large for all groups except P, XPREP suggests that the lattice is P (Figure 7.12). Press [Enter] to accept.

Figure 7.12 —Lattice statistics

```
++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++
+  XPREP - Reciprocal space exploration  - Version 2008/2 for Windows  +
+  COPYRIGHT(c) 2008 Bruker-AXS                  All Rights Reserved  +
++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++++

Screen size: 1600 x 1200
Window size:  640 x 1099
Font size:      8 x   16  (  125 x  150 )
Number of colors:    256

When xprep is started without a filename on the command line, the filename
is prompted for and then the type of data (SHELX, SCALEPACK, XDS or XENGEN)
requested.  To generate ideal data, a SHELX .ins or .res file, if necessary
made from a PDB file using SHELXPRO or XPRO, should be given.

'xprep name' reads a SHELX HKLF 4 format file name.hkl, then tries to find
name.spin or name.p4p to extract the cell dimensions and their esds.
'xprep name1 name2' reads name1.hkl and name2.p4p (or name2.spin).

-Ln on the command line allocates space for 1000000n data (default n=4).

** Data multiplied by   0.1000  to bring onto reasonable scale **

   8138 Reflections read from file ylid_0m.hkl
  Mean (I/sigma) =   25.72


Lattice exceptions: P      A      B      C      I      F     Obv    Rev    All

N (total) =          0   4072   4063   4093   4075   6114   5423   5422   8138
N (int>3sigma) =     0   3800   3757   3817   3749   5687   5033   5033   7552
Mean intensity =   0.0   25.3   23.9   23.3   25.4   24.2   24.7   24.6   24.8
Mean int/sigma =   0.0   26.0   24.7   25.6   25.6   25.4   25.7   25.6   25.7

Lattice type [P, A, B, C, I, F, O(obv.), R(rev. rhomb. on hex. axes)]

Select option [P]:
```

2. XPREP determines the reduced cell based on the lattice entered above. Since the lattice was primitive and the magnitudes of the cell dimensions were proper (a < b < c), the original and reduced cells are the same (Figure 7.13). Press [Enter] to search for a higher symmetry cell.

Figure 7.13 —Reduced cell

```
Current dataset: ylid_0m.hkl                    Wavelength: 0.71073 Chiral: ?
------------------------------------------------------------------------------
Original cell:    5.961   9.037  18.390   90.00   90.00   90.00    Vol      990.6
         Esds:    0.000   0.000   0.001    0.00    0.00    0.00        Lattice: P
------------------------------------------------------------------------------
Current cell:     5.961   9.037  18.390   90.00   90.00   90.00    Vol      990.6
------------------------------------------------------------------------------
Matrix: 1.0000  0.0000  0.0000  0.0000  1.0000  0.0000  0.0000  0.0000  1.0000
------------------------------------------------------------------------------


[D] Read, modify or merge DATASETS          [C] Define unit-cell CONTENTS
[P] Contour PATTERSON sections              [F] Set up shelxtl FILES
[H] Search for HIGHER metric symmetry       [R] RECIPROCAL space displays
[S] Determine or input SPACE GROUP          [U] UNIT-CELL transformations
[A] Absorption, powder, SIR, SAD, MAD etc.  [T] Change TOLERANCES
[M] Test for MEROHEDRAL TWINNING            [O] Self-rotation function
[L] Reset LATTICE type of original cell     [Q] QUIT program

Select option [H]:
```

3. For the YLID, no higher symmetry cell is found. The program has determined that the YLID crystal has an orthorhombic primitive lattice (Figure 7.14). Press [Enter] to accept.

Figure 7.14 —Higher-symmetry cells

```
Determination of reduced (Niggli) cell

Transformation from original cell (HKLF-matrix):
   1.0000  0.0000  0.0000    0.0000  1.0000  0.0000    0.0000  0.0000  1.0000

Unitcell:       5.961    9.037   18.390   90.00   90.00   90.00

Niggli form:    a.a =     35.53      b.b =     81.67      c.c =    338.20
                b.c =      0.00      a.c =      0.00      a.b =      0.00

Search for higher METRIC symmetry
Identical indices and Friedel opposites combined before calculating R(sym)

------------------------------------------------------------------------------
Option A: FOM = 0.000 deg.   ORTHORHOMBIC P-lattice   R(sym) = 0.012 [   3110]
Cell:    5.961   9.037  18.390   90.00   90.00   90.00    Volume:       990.64
Matrix: 1.0000  0.0000  0.0000  0.0000  1.0000  0.0000  0.0000  0.0000  1.0000
------------------------------------------------------------------------------
Option B retains original cell

Select option [A]: 
```

4. The next logical step is to determine the space group. XPREP suggests this (Figure 7.15). Press [Enter] to determine the space group.

Figure 7.15 —Determine the space group

```
Current dataset: ylid_0m.hkl                    Wavelength: 0.71073 Chiral: ?
------------------------------------------------------------------------------
Original cell:   5.961   9.037  18.390   90.00   90.00   90.00    Vol      990.6
         Esds:   0.000   0.000   0.001    0.00    0.00    0.00       Lattice: P
------------------------------------------------------------------------------
Current cell:    5.961   9.037  18.390   90.00   90.00   90.00    Vol      990.6
------------------------------------------------------------------------------
Matrix: 1.0000  0.0000  0.0000  0.0000  1.0000  0.0000  0.0000  0.0000  1.0000
------------------------------------------------------------------------------
Crystal system: Orthorhombic            Lattice: P
------------------------------------------------------------------------------


[D] Read, modify or merge DATASETS          [C] Define unit-cell CONTENTS
[P] Contour PATTERSON sections              [F] Set up shelxtl FILES
[H] Search for HIGHER metric symmetry       [R] RECIPROCAL space displays
[S] Determine or input SPACE GROUP          [U] UNIT-CELL transformations
[A] Absorption, powder, SIR, SAD, MAD etc.  [T] Change TOLERANCES
[M] Test for MEROHEDRAL TWINNING            [O] Self-rotation function
[L] Reset LATTICE type of original cell     [Q] QUIT program

Select option [S]: 
```

5. If the space group is known or if the compound is known to be chiral, enter that information (Figure 7.16). Generally, it is sufficient to press [Enter] to start the space group determination.

Figure 7.16 —Space group options

```
[S] Determine SPACE GROUP
[C] Must be CHIRAL (sample is optically active)
[N] NOT NECESSARILY chiral (eg. may be racemate)
[I] INPUT known space group
[E] EXIT to main menu  or  [Q] QUIT program

Select option [S]: 
```

6. XPREP has chosen the crystal system [O] (Figure 7.17). Press [Enter] to accept.

Figure 7.17 —Choose the crystal system

```
[A] Triclinic,  [M] Monoclinic,  [O] Orthorhombic,  [T] Tetragonal,
[H] Trigonal/Hexagonal,  [C] Cubic  or  [E] EXIT

Select option [O]: 
```

7. XPREP has chosen the crystal lattice [P] (Figure 7.18). Press [Enter] to accept.

Figure 7.18 —Choose the lattice type

```
Lattice exceptions: P      A      B      C      I      F     Obv    Rev    All

N (total) =          0   5634   5622   5620   5623   8438   7497   7501  11249
N (int>3sigma) =     0   5157   5089   5194   5096   7720   6834   6834  10243
Mean intensity =   0.0  224.9  214.3  211.3  225.4  216.9  223.9  224.6  220.3
Mean int/sigma =   0.0   20.7   19.7   20.9   20.5   20.4   20.5   20.5   20.5

Lattice type [P, A, B, C, I, F, O(obv.), R(rev. rhomb. on hex. axes)]

Select option [P]:
```

8. XPREP evaluates the data and looks at the systematic absences for all possible glide planes and screw axes (Figure 7.19). These are displayed across the middle of the figure. By examining the number of reflections with I > 3 sigma(I), the mean intensities, and the mean int/sigma, which should all be very small for a systematic absence, XPREP derives a suggested space group, P2(1)2(1)2(1). Press [Enter] to accept.

Figure 7.19 —Systematic absences and a suggested space group

```
Mean |E*E-1| = 0.687 [expected .968 centrosym and .736 non-centrosym]


Systematic absence exceptions:

        b--   c--   n--  21--   -c-   -a-   -n-  -21-   --a   --b   --n  --21

N       457   455   460    16   360   374   358    19   233   230   223    17
N I>3s  388   394   386     0   281   282   229     0   192   203   171     0
<I>   201.1 300.0 317.2   0.5 449.8 377.8 201.2   0.5 290.7 311.3 277.0   0.6
<I/s>  22.1  22.6  23.6   0.4  23.9  21.5  15.9   0.6  20.8  22.7  21.7   0.6


Identical indices and Friedel opposites combined before calculating R(sym)

Option  Space Group  No.  Type  Axes  CSD  R(sym) N(eq)  Syst. Abs.   CFOM

[A] P2(1)2(1)2(1)  # 19  chiral   1  5917  0.015   3394  0.6 / 15.9   0.89

Select option [A]:
```

7.2.2 Examine Reflection Statistics

1. After determining the space group, XPREP returns to the general menu. This time, D is chosen to evaluate the data set. Press [Enter] to accept.
2. There are multiple choices for data manipulation (Figure 7.20). “S” chooses a display of statistics.

Figure 7.20 —Data manipulation menu

```
Index   # Data   Filename or Source of Data

   1    11249    ylid_0m.hkl <- current dataset

[M] Sort-MERGE current data (no scaling)     [C] Change CURRENT dataset
[L] LEAST-SQUARES scale and merge datasets   [W] WRITE dataset to file
[I] INCLUDE Rfree flags from another file    [R] READ in another dataset
[S] Display intensity STATISTICS             [D] DELETE stored dataset
[F] FACE-indexed absorption corrections      [P] PSI-scan absorption corr.
[T] Copy file, TRANSFORM hkl and cosines     [A] MAD, SAD, SIR or SIRAS
[H] Apply HIGH/low resolution cutoffs        [N] NORMALIZE/scale sigmas
[G] Generate simulated powder diagrams       [U] Anisotropic scaling
[Z] Expand data to triclinic                 [E] EXIT to main menu
[X] Parsons' Q values and Flack x parameter  [Q] QUIT program

Select option [S]:
```

3. The data can be merged in several ways. Choose the “Merge ALL equivalents including Friedel opposites” option [A] (Figure 7.21). Press [Enter] to accept.

NOTE: This merge will not average the reflections in the final data file. It is only for the calculation of statistics.

Figure 7.21 —Merge data menu

```
Current dataset: ylid_0m.hkl

[R] Define resolution ranges (currently selected automatically)
[P] Output R(pim) rather than R(sigma)
[N] Do NOT merge reflections
[I] Merge IDENTICAL indices only
[S] Merge SYMMETRY equivalents (but not Friedel opposites)
[A] Merge ALL equivalents (including Friedel opposites)
[E] EXIT to merge datasets menu
[Q] QUIT program

Select option [A]:
```

4. A table of statistics appears (Figure 7.22). Examine the data presented. Is the completion near 100%? Is the redundancy good? Are R_{int} and R_{sigma} small and increasing smoothly from top to bottom? In particular, look at the last two lines which compare all of the data with the high-resolution data. The completion should be near 100% for both the high-resolution shell and the complete data set. The redundancy and Rint should be similar for the two. The Mean Intensity and the Mean I/sigma(I) will usually be quite different. The Mean I/sigma(I) for the high-resolution data should be greater than 3.0.

Figure 7.22 —Intensity statistics

```
 Resolution  #Data #Theory %Complete Redundancy Mean I  Mean I/s  Rint  Rsigma

  Inf - 2.23     75     77     97.4      7.08   1377.5  103.56  0.0177  0.0085
2.23 - 1.73      75     75    100.0      9.09    575.4  102.60  0.0171  0.0084
1.73 - 1.47      79     79    100.0      9.28    447.7  105.41  0.0175  0.0084
1.47 - 1.32      81     81    100.0      9.62    246.1   89.44  0.0191  0.0099
1.32 - 1.21      76     76    100.0      9.03    211.7   82.63  0.0212  0.0104
1.21 - 1.14      77     77    100.0      9.08    227.9   78.60  0.0224  0.0108
1.14 - 1.08      79     79    100.0      8.56    124.2   59.78  0.0284  0.0132
1.08 - 1.02      86     86    100.0      7.77     98.5   53.04  0.0298  0.0159
1.02 - 0.98      78     78    100.0      7.78    105.3   51.83  0.0321  0.0161
0.98 - 0.94      95     95    100.0      7.80     71.5   43.72  0.0374  0.0200
0.94 - 0.90     103    103    100.0      7.20     62.5   35.89  0.0402  0.0231
0.90 - 0.87      89     89    100.0      6.63     46.5   30.32  0.0513  0.0289
0.87 - 0.84      99     99    100.0      6.74     38.5   27.15  0.0624  0.0327
0.84 - 0.82      89     89    100.0      6.66     43.1   26.73  0.0569  0.0313
0.82 - 0.80      82     82    100.0      6.40     30.6   21.75  0.0685  0.0412
0.80 - 0.78      93     93    100.0      6.29     30.8   21.51  0.0735  0.0418
0.78 - 0.76     100    100    100.0      6.14     27.1   18.60  0.0850  0.0488
0.76 - 0.74      26     28     92.9      4.18     28.3   15.58  0.0699  0.0560
-------------------------------------------------------------------------------
0.84 - 0.74     425    427     99.5      6.24     33.2   22.11  0.0685  0.0401
  Inf - 0.74   1482   1486     99.7      7.57    199.6   52.87  0.0225  0.0120

Merged [A],  lowest resolution =  9.18 Angstroms,    363 outliers downweighted


Enter <CR> to continue
```

5. Press [Enter] to continue.

6. Press [Enter] again to exit to the main XPREP menu.

7.2.3 Prepare an Output File

1. In the general menu, choose [C] to define the unit cell contents.

   A window opens displaying the current formula, Z, the density, and the atomic volume (see Figure 7.23). In this example, the formula is incorrect and Z has been set to six to try to achieve a reasonable density and atomic volume. Since this formula is incorrect, it must be modified now. The correct chemical formula for the ylid crystal is $C_{11}H_{10}O_2S$.

2. At “Select option”, do not accept the default answer of E. Type in [F] to enter a new formula.

3. In response to the question “Enter Formula,” type the correct formula [C11 H10 O2 S1] and press [Enter].

4. Check that the information is correct. Check that Z seems reasonable for the space group, that the density is as expected (1.1 to 1.4 for organic molecules, higher for inorganic compounds), and that the atomic volume is around 17 or 18. Significant variation from the expected values may indicate an incorrect molecular formula or missing counter ions or solvates. The values at the bottom of the window look fine for the ylid. Press [Enter] to accept.

Figure 7.23 —Defining and checking the unit cell contents

```
Current dataset: ylid_0m.hkl                    Wavelength: 0.71073 Chiral: ?
-------------------------------------------------------------------------------
Original cell:   5.957   9.027  18.366   90.00   90.00   90.00   Vol      987.7
         Esds:   0.001   0.001   0.002    0.00    0.00    0.00       Lattice: P
-------------------------------------------------------------------------------
Current cell:    5.957   9.027  18.366   90.00   90.00   90.00   Vol      987.7
-------------------------------------------------------------------------------
Matrix: 1.0000  0.0000  0.0000  0.0000  1.0000  0.0000  0.0000  0.0000  1.0000
-------------------------------------------------------------------------------
Crystal system: Orthorhombic  Space group: P2(1)2(1)2(1)  # 19 [chi]   Laue: 3
-------------------------------------------------------------------------------
Formula: C11 H10 O2 S1                                  Formula wt:   206.25
Z:  4.00  Density:  1.387  At.vol:  17.6  F(000):    432.00  Mu[mm-1]:    0.30
-------------------------------------------------------------------------------

[D] Read, modify or merge DATASETS          [C] Define unit-cell CONTENTS
[P] Contour PATTERSON sections              [F] Set up shelxtl FILES
[H] Search for HIGHER metric symmetry       [R] RECIPROCAL space displays
[S] Determine or input SPACE GROUP          [U] UNIT-CELL transformations
[A] Absorption, powder, SIR, SAD, MAD etc.  [T] Change TOLERANCES
[M] Test for MEROHEDRAL TWINNING            [O] Self-rotation function
[L] Reset LATTICE type of original cell     [Q] QUIT program

Select option [Q]: f

Output file name (without extension) [ylid_0m]:
```

5. The next default action for XPREP is to write out the files necessary for the structure solution process (see Figure 7.24). Press [Enter] to accept.

Figure 7.24 —Requesting output files

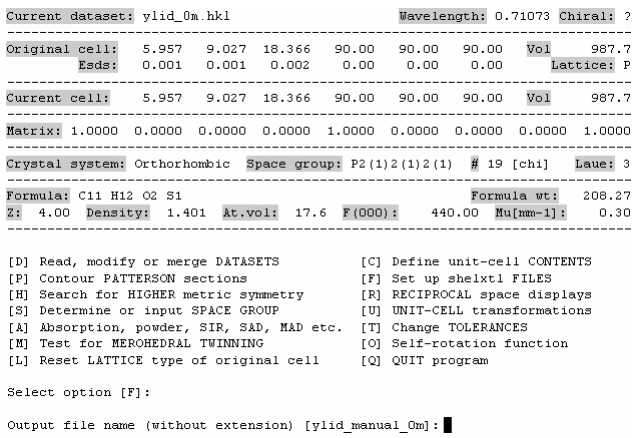
```
Current dataset: ylid_0m.hkl                    Wavelength: 0.71073 Chiral: ?
-------------------------------------------------------------------------------
Original cell:   5.957   9.027  18.366   90.00   90.00   90.00   Vol      987.7
         Esds:   0.001   0.001   0.002    0.00    0.00    0.00       Lattice: P
-------------------------------------------------------------------------------
Current cell:    5.957   9.027  18.366   90.00   90.00   90.00   Vol      987.7
-------------------------------------------------------------------------------
Matrix: 1.0000  0.0000  0.0000  0.0000  1.0000  0.0000  0.0000  0.0000  1.0000
-------------------------------------------------------------------------------
Crystal system: Orthorhombic  Space group: P2(1)2(1)2(1)  # 19 [chi]   Laue: 3
-------------------------------------------------------------------------------
Formula: C11 H12 O2 S1                                  Formula wt:   208.27
Z:  4.00  Density:  1.401  At.vol:  17.6  F(000):    440.00  Mu[mm-1]:    0.30
-------------------------------------------------------------------------------

[D] Read, modify or merge DATASETS          [C] Define unit-cell CONTENTS
[P] Contour PATTERSON sections              [F] Set up shelxtl FILES
[H] Search for HIGHER metric symmetry       [R] RECIPROCAL space displays
[S] Determine or input SPACE GROUP          [U] UNIT-CELL transformations
[A] Absorption, powder, SIR, SAD, MAD etc.  [T] Change TOLERANCES
[M] Test for MEROHEDRAL TWINNING            [O] Self-rotation function
[L] Reset LATTICE type of original cell     [Q] QUIT program

Select option [F]:
```

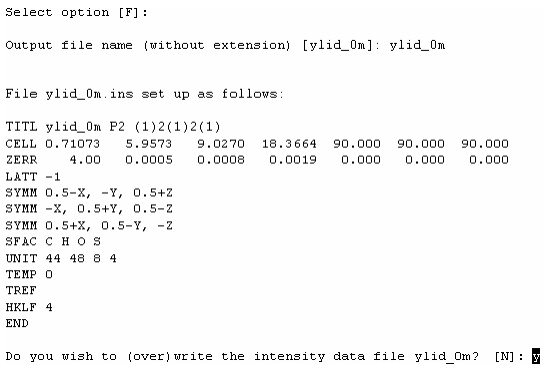
6. The program asks for an output file name. Press [Enter] to accept.

Figure 7.25 —Output file name



7. After entering the file name or pressing [Enter] to accept the default file name, an input file for the structure solution module is created and displayed on the screen, and the program asks, “Do you wish to (over)write the intensity data file ylid_0m.hkl?” Since the file name has been changed, this question must be answered with a “y” (see Figure 7.26).

Figure 7.26 —The input file for structure solution



7.2.4 Quit XPREP

1. At XPREP’s main menu, choose Q (or press [Enter] if Q is the default) to exit the program.

7.3 Create Simulated Precession Images

The Precession Images module provides an undistorted view of layers of the reciprocal lattice. The module generates simulated precession images by finding the appropriate pixels in a series of ".sfrm" images. You must specify the images to examine and the zones to calculate.

1. In the Task Bar's "Examine" category, click **Precession Images**.

   The Precession Images module opens.

2. Select the runs for image synthesis by clicking the **Browse** button beside the "Images From:" field.

   A "Select Runs" dialog opens.

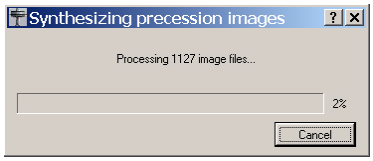
3. Choose the sets of images that you want to use for the calculation by activating and deactivating the checkboxes. Click **OK**.

4. Check the default input values. Modify as needed, and click **Calculate**.

   Calculating more zones does not significantly increase the calculation time. Reducing the resolution may speed the calculations slightly. The thickness defines the range of pixels above and below the requested range. For example, if the 0kl zone is requested with a thickness of 0.1, then the simulation will use all pixels that have -0.1 < h < 0.1, and any value (including fractional values) for k and l.

   A progress bar appears (Figure 7.27).

Figure 7.27 —Progress bar



   After a short time (depending on the speed of the computer and the number of ".sfrm" files read), the simulated precession image will appear on the screen.

5. Display other planes by clicking on the calculated images in the bottom right of the Task Display Area, or by browsing as usual with the View Images module.

6. Use the simulated patterns to check space group symmetry (Figure 7.28, Figure 7.29, and Figure 7.30) and to find signs of twinning (Figure 7.31).

Figure 7.28 —Example 0kl plane

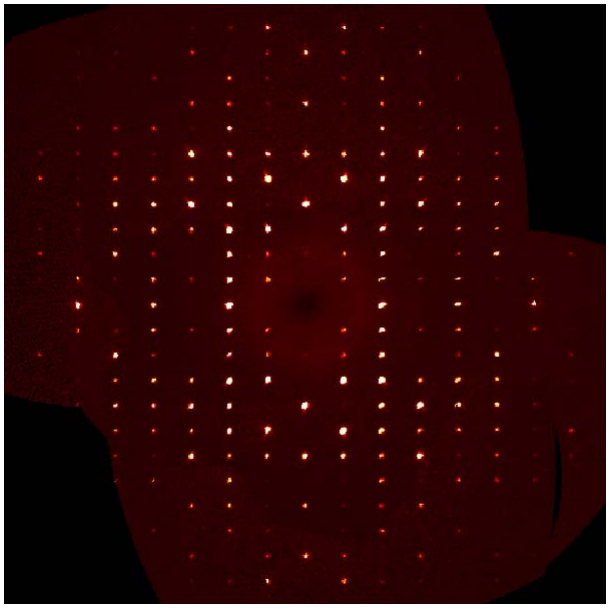


Figure 7.29 —Example h0l plane

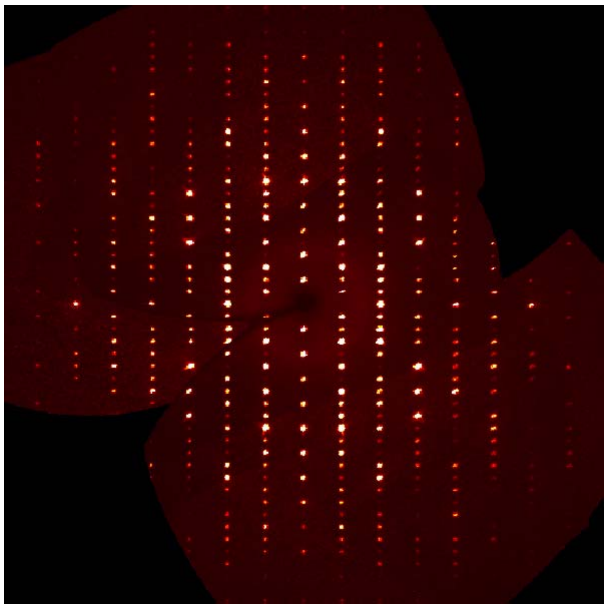


Figure 7.30 —Example hk0 plane

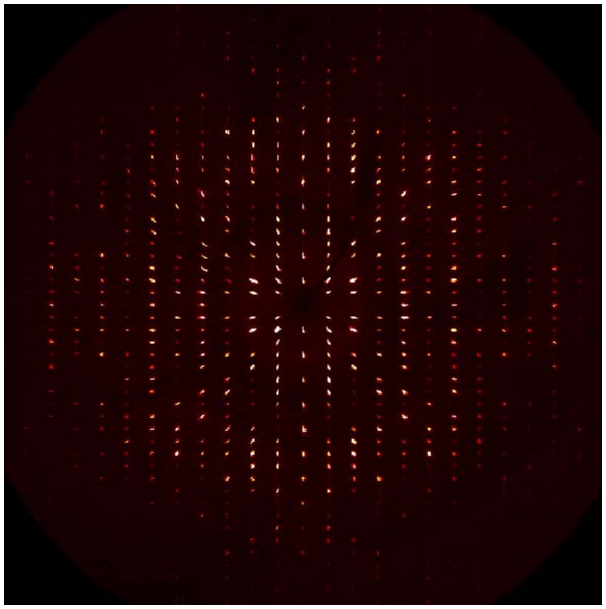
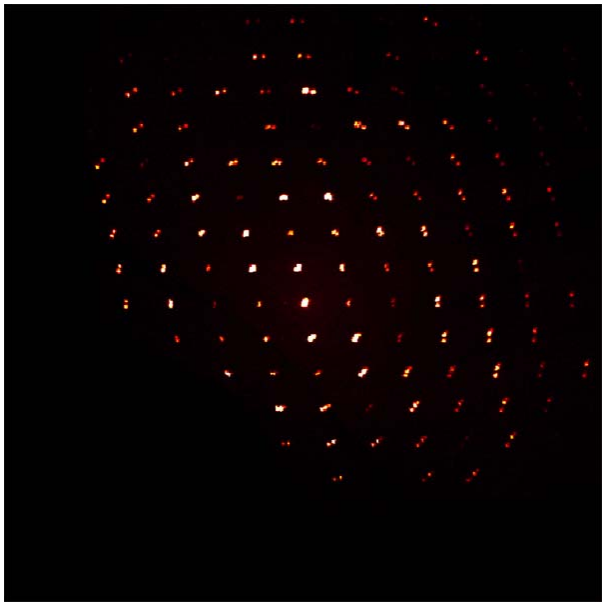


Figure 7.31 —A plane from a rotationally-twinned crystal, showing the two lattices



8 Structure Solution and Refinement

The .ins and .hkl files (output by the Space Groups and Statistics module or, alternatively, XPREP) are all that is required to begin the structure solution and refinement process. The various steps of solving and refining the structure are carried out using the Structure Solution and Structure Refinement modules.

8.1 Using AUTOSTRUCTURE

8.1.1 Before you Begin

Required Files

Before using AUTOSTRUCTURE, make sure that you have:

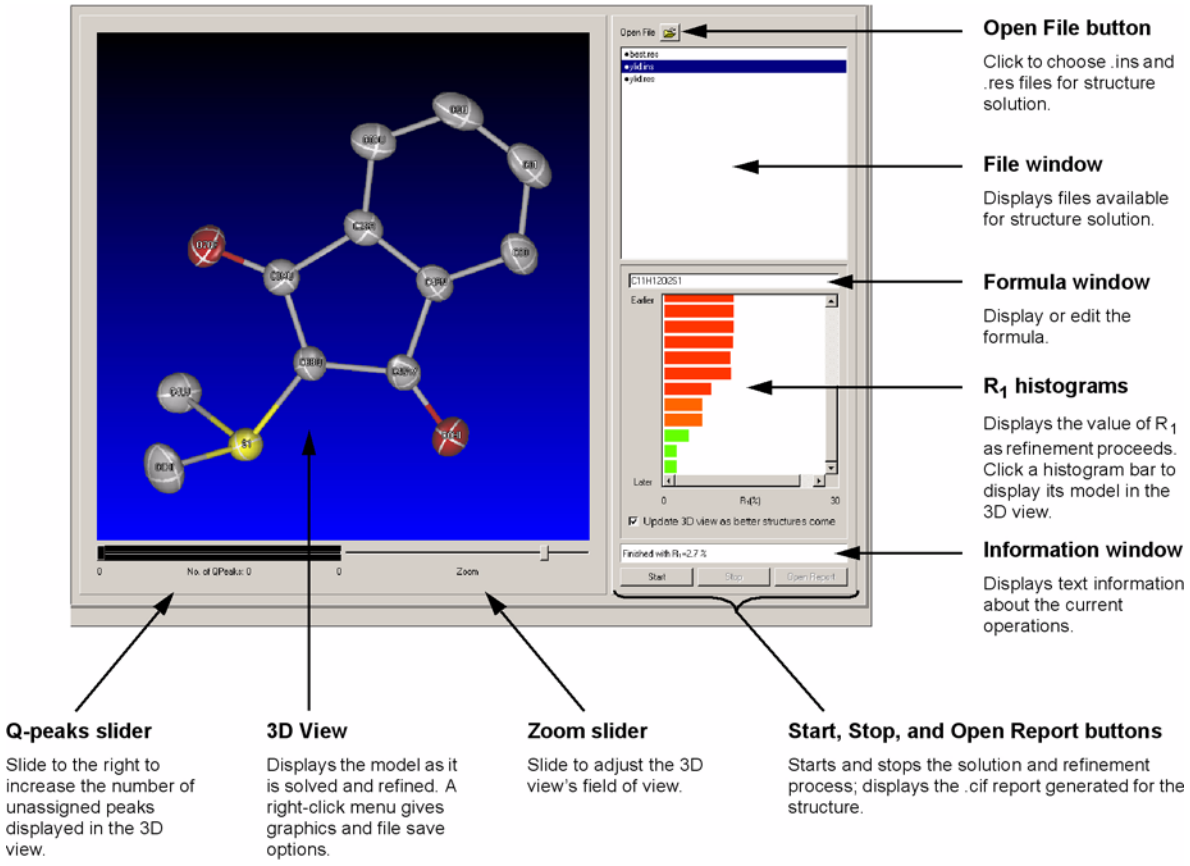
- Unit cell and formula unit (.ins or .res file)
- Set of indexed reflections (.hkl file)

8.1.2 Solving the Structure

1. In the Task Bar's "Solve Structure" category, click the **AUTOSTRUCTURE** icon .

   The AUTOSTRUCTURE module opens.

Figure 8.1 — AUTOSTRUCTURE GUI



2. Click the **Open File** icon in the top right-hand corner of the module.

   A "Choose File" dialog window opens.

3. Navigate to an .ins or .res file.

   Only the unit cell and formula information from this file will be used. There should be an .hkl file in the same directory containing the list of indexed reflections.

4. Click **Open**.

   The "Choose File" dialog window closes, the information gathered from the file is displayed, and the **Start** button becomes enabled.

NOTE: The displayed formula is the formula unit (derived from the .ins file's `UNIT` line and Z), but the formula unit field is editable.

8.1.3 Automatically Solving and Refining the Structure

1. Click **Start**.

   The **Stop** button becomes enabled, and AUTOSTRUCTURE begins solving and refining the structure.

   As structure solution proceeds, the lower right-hand window displays progress information. The R_1 histogram will update regularly as the solution improves. The text window below the R_1 histogram displays the current phase: “solving…”, “refining…”, etc., as well as any errors that may occur.

NOTE: Clicking **Stop** will not stop the auto-solution immediately; the process will stop after the display’s next update.

   When the auto-solution process is finished, the final model will appear in the 3D view and the final R_1 value will appear in the text window.

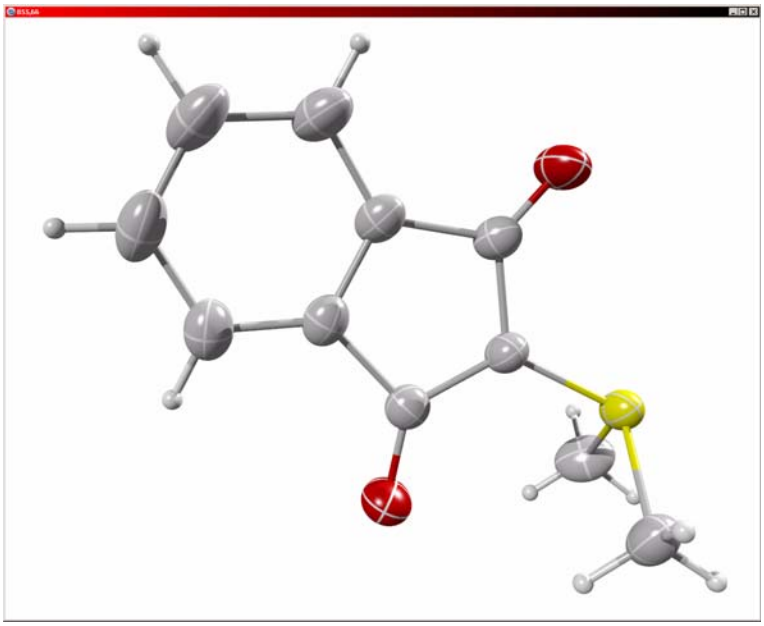
2. Save the image by right-clicking in the 3D view. Choose the desired format and resolution under **Save Image**.

   The following image formats are available:

   - Bitmap (*.bmp)
   - JPEG (*.jpg)
   - PNG Image (*.png)

NOTE: The molecular model is also available as a scene file for the free POV-Ray raytracing program. Choose **POV-Ray** from the right-click menu.

Figure 8.2 — Structure rendered in POV-Ray

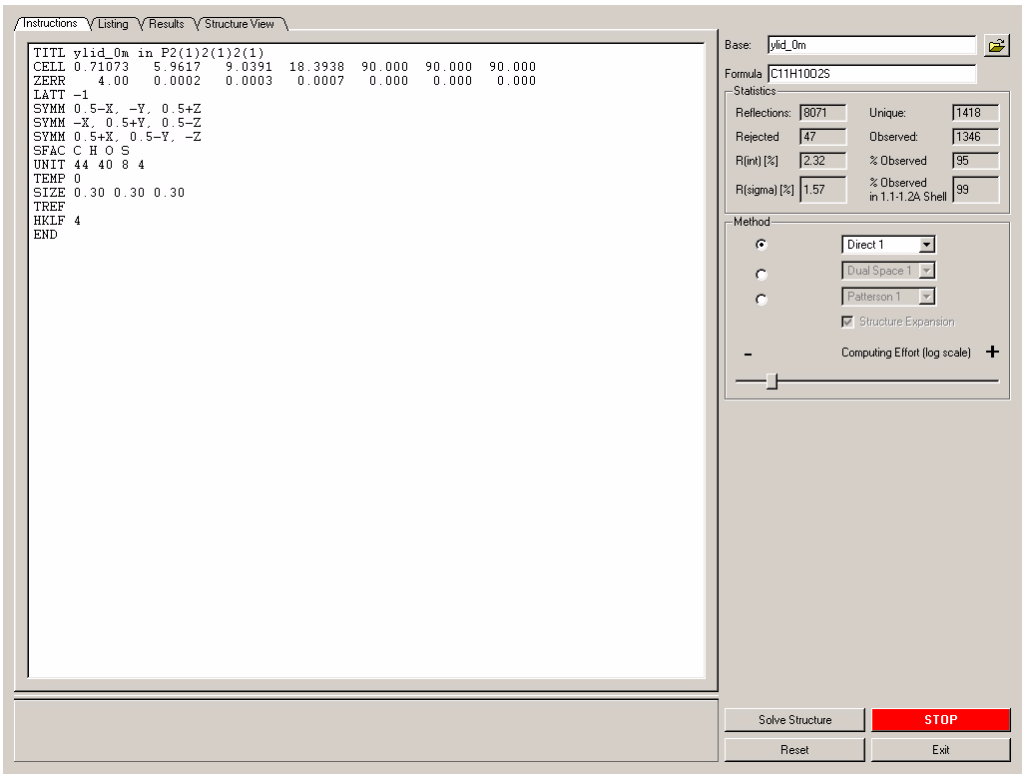


8.2 Solving the Structure with the Structure Solution Module

1. In the Task Bar's "Solve Structure" category, click the **Structure Solution** icon .

   The Structure Solution module opens.

Figure 8.3 — Structure Solution module: initial view

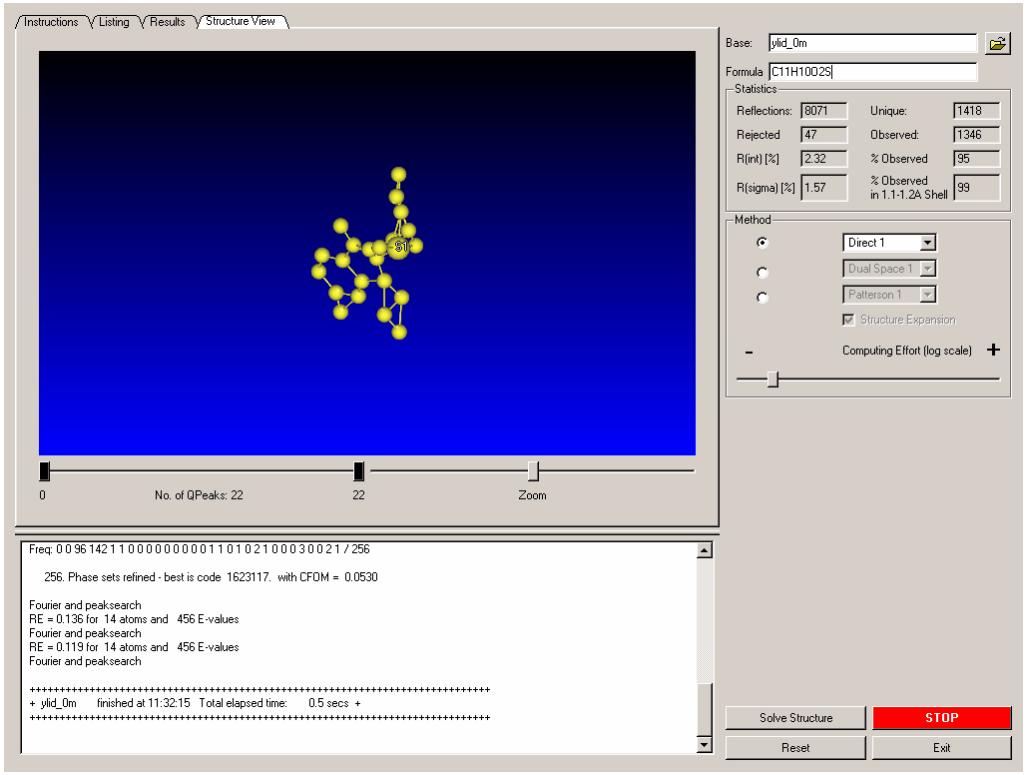


2. Check that there is a reasonable formula in the "Formula" window.
   - If you have a correct formula in the formula field, then you are ready to start a normal direct methods run.
   - If the formula is incorrect, change it and press [Enter]. The input file will be updated.

3. Click **Solve Structure** to start the direct methods calculations.

   The upper window displays the Q-peaks of the initial model, and the text area displays the progress of the calculations.

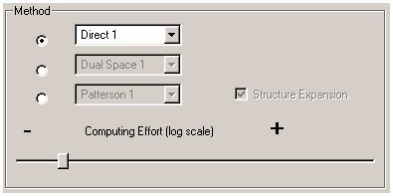
Figure 8.4 — Structure Solution module output



8.2.1 Direct Methods Options

The “Method” area’s first drop-down menu (Figure 8.5) gives three preset choices for structure solution using direct methods.

Figure 8.5 — “Method” area



In most cases the default values will give a good initial model, but several other options are available for more difficult problems. Chose the solution method by clicking the appropriate radio button in the “Method” area.

Table 8.1 — Direct methods presets

Preset	Description
Direct 1	Standard settings, which should be appropriate for a wide range of circumstances. Sets up a default run with a simple `TREF` instruction.
Direct 2	Brute-force method with a higher number of direct methods attempts. Sets up an extended run with `TREF 10000`. By adding `10000` to the `TREF` command, more attempts are made to determine a solution. This will take more time, but if you see a good solution in the output window it is possible to stop at that point by clicking **Stop**. Good solutions typically have a CFOM of 0.06 or less.
Direct 3	Brute-force method with even more direct methods attempts. Sets up an extreme run with two instructions: `ESEL 1.0` and `TREF 100000`. By adding `ESEL 1.0`, more reflections are used in the solution process. Increasing the number of tries in the `TREF` command to `100000` runs (until ended by clicking **Stop**) is particularly useful for acentric triclinic structures and for pseudosymmetric structures.

For direct methods calculations, good figures of merit are near 1.0 for Sigma-1 and M(abs) and less than 0.06 for R_{alpha} and the CFOM. Structures can be solved with figures of merit that deviate from these numbers, but they may require more effort.

If the initial model looks reasonable, click **OK** in the output display and **Exit** in the “Structure Solution” module. The module automatically switches to the “Display” tab and displays the initial solution.

8.2.2 Dual-Space Methods Options

Two predefined options are available for the Dual-Space method. Dual-Space methods are effective for larger organic molecules and polypeptides.

More information on Dual-Space methods and their use within APEX2 is available from the “What’s This?” Help function.

8.2.3 Patterson Methods Options

Two predefined options are available for Patterson methods. Patterson methods are effective for finding heavy-atom positions. In general, Patterson methods favor a small number of strong scatterers.

More information on Patterson methods and their use within APEX2 is available from the “What’s This?” Help function.

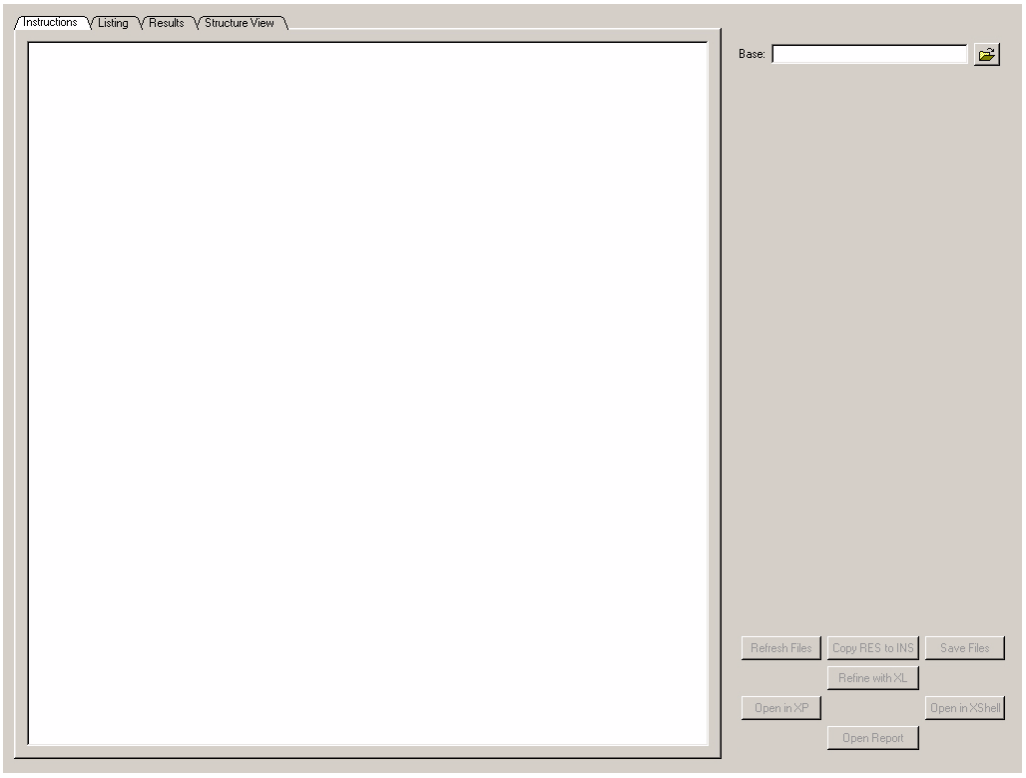
8.3 Refining the Structure with the Structure Refinement Module

The structure solution step produced statistics indicating that a solution had been found. The real proof, however, is in the initial model that is produced. XSHELL provides the tools to view and refine the model. A quick glance at the results of the direct methods run is often all that is needed to see that the results make chemical sense. Control of the refinement process is quite straightforward using XSHELL.

1. Under the Task Bar's "Refine Structure" category, click the **Structure Refinement** icon .

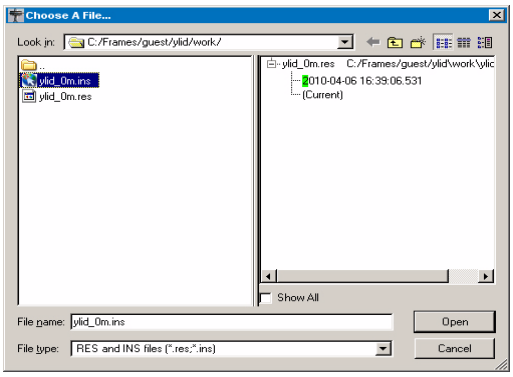
   The "Structure Refinement" module opens. It contains tabbed sections for viewing the instructions file, listing file, results file, and a 3D model of the structure.

Figure 8.6 — "Structure Refinement" module initial view



2. Click the **Browse** icon on the right-hand side of the “Base:” field, and select the desired .ins file in the “Choose a File...” dialog.

Figure 8.7 — “Choose a File...” dialog



3. Click **Open**.

   The contents of the .ins file and any other files with the same base name are displayed under their respective tabs. Also, the six buttons in the lower right-hand corner of the module become available.

Table 8.2 — “Structure Refinement” module buttons

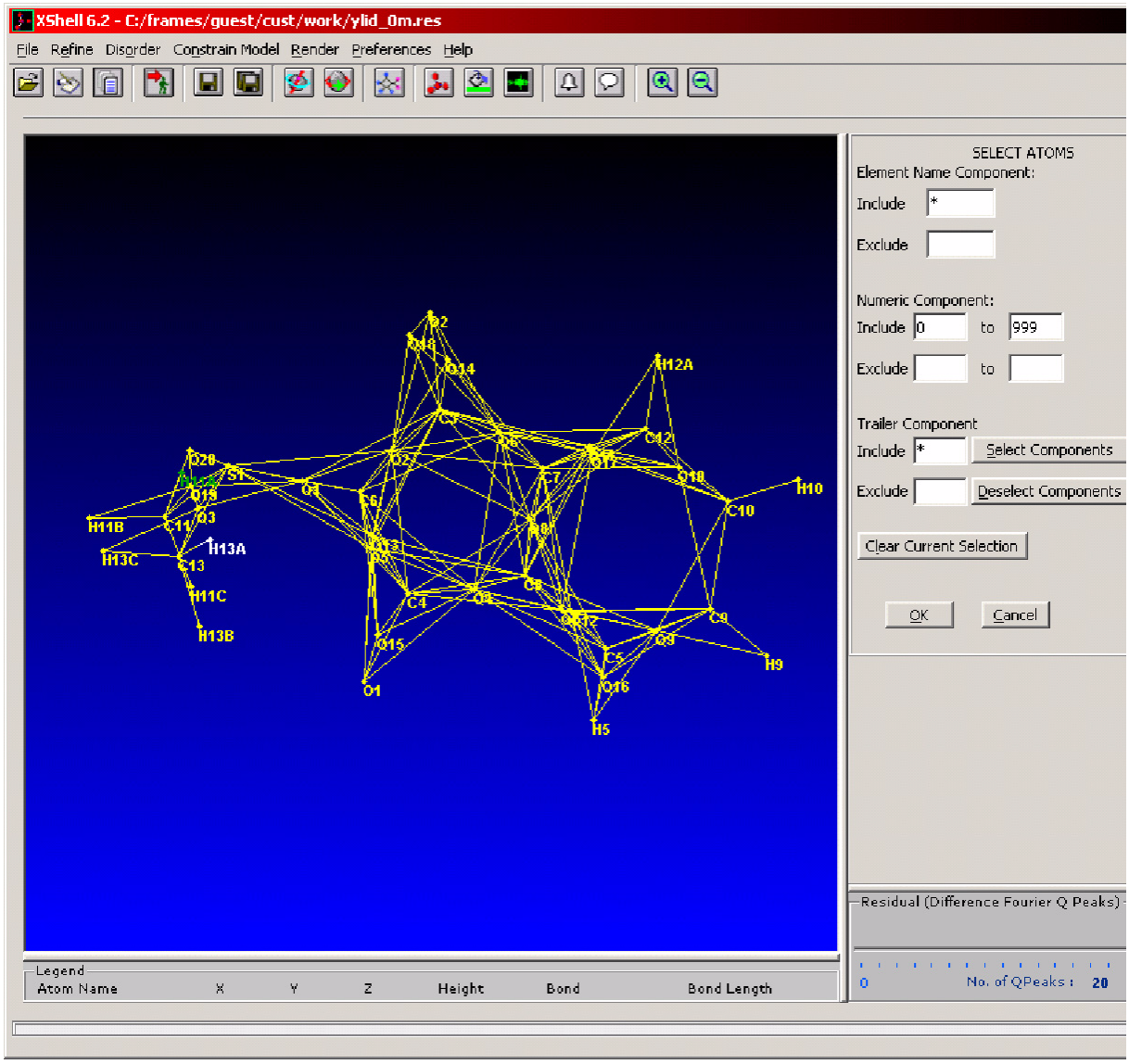
Button	Function
Refresh Files	Refresh the tabs using the absolute latest contents of all the files.
Copy RES to INS	Copy the contents of the results file to the instructions file.
Save Files	Save any changes you have made to the files.
Refine with XL	Refine the instruction file using XL.
Open in XP	Open XP for editing the results file.
Open in XSHELL	Open the selected file in XSHELL.

8.3.1 Refining the Structure in XSHELL

1. Click **Open in XSHELL**.

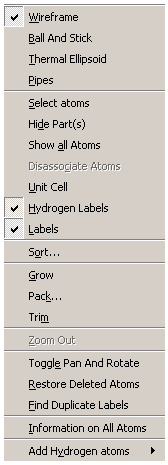
   The XSHELL Main Window appears.

Figure 8.8 — Initial view of the ylid molecule



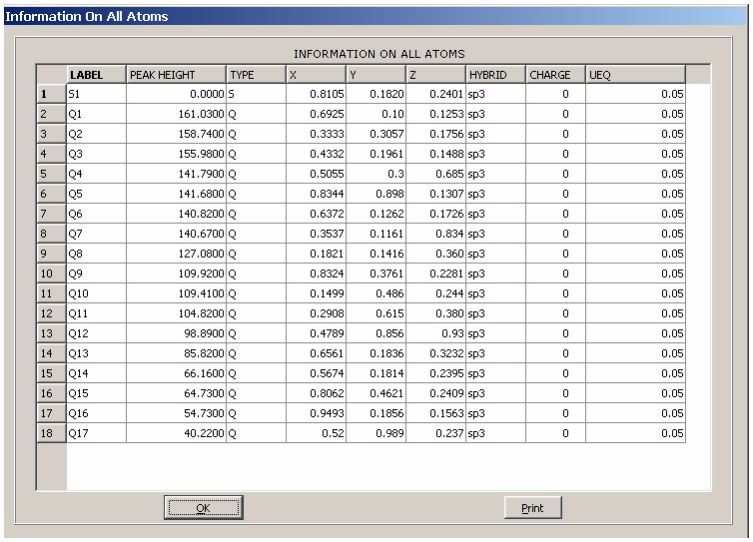
2. Right-click in the background to open a menu with image display options.

Figure 8.9 — Right-click menu



3. Choose **Information on All Atoms** to open a window displaying a list of the peaks and their heights (Figure 8.10). The drop in height between Q13 (the last real peak) and Q14 (the first noise peak) is typical of a correct solution. Click **OK** to close this window.

Figure 8.10 —"Information on all atoms" window



	LABEL	PEAK HEIGHT	TYPE	X	Y	Z	HYBRID	CHARGE	UEQ
1	S1	0.0000	S	0.8105	0.1820	0.2401	sp3	0	0.05
2	Q1	161.0300	Q	0.6925	0.10	0.1253	sp3	0	0.05
3	Q2	158.7400	Q	0.3333	0.3057	0.1756	sp3	0	0.05
4	Q3	155.9800	Q	0.4332	0.1961	0.1488	sp3	0	0.05
5	Q4	141.7900	Q	0.5055	0.3	0.685	sp3	0	0.05
6	Q5	141.6800	Q	0.8344	0.898	0.1307	sp3	0	0.05
7	Q6	140.8200	Q	0.6372	0.1262	0.1726	sp3	0	0.05
8	Q7	140.6700	Q	0.3537	0.1161	0.834	sp3	0	0.05
9	Q8	127.0800	Q	0.1821	0.1416	0.360	sp3	0	0.05
10	Q9	109.9200	Q	0.8324	0.3761	0.2281	sp3	0	0.05
11	Q10	109.4100	Q	0.1499	0.486	0.244	sp3	0	0.05
12	Q11	104.8200	Q	0.2908	0.615	0.380	sp3	0	0.05
13	Q12	98.8900	Q	0.4789	0.856	0.93	sp3	0	0.05
14	Q13	85.8200	Q	0.6561	0.1836	0.3232	sp3	0	0.05
15	Q14	66.1600	Q	0.5674	0.1814	0.2395	sp3	0	0.05
16	Q15	64.7300	Q	0.8062	0.4621	0.2409	sp3	0	0.05
17	Q16	54.7300	Q	0.9493	0.1856	0.1563	sp3	0	0.05
18	Q17	40.2200	Q	0.52	0.989	0.237	sp3	0	0.05

4. At the bottom right of the main window is a slider which is used for deselecting peaks. The arrow can be moved by clicking and dragging the arrow or by clicking on either side of the arrow. Clicking to the left of the arrow (i.e., where the mouse cursor is pointed in Figure 9.9) removes peaks. Slide the pointer down while watching the molecular display. Reduce the number of Q peaks until you find a reasonable model.

Figure 8.11 — Q peaks slider

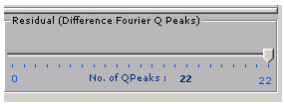


Figure 8.12 — Image after the number of Q peaks was changed to 15

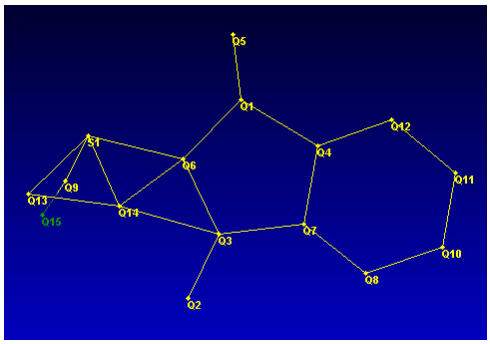
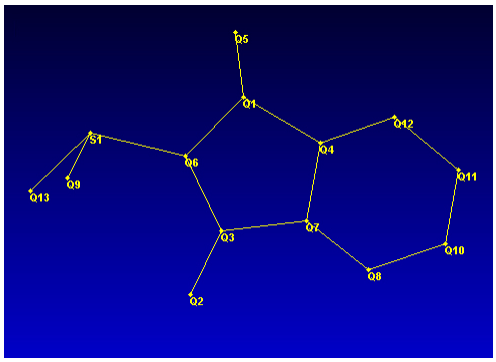


Figure 8.13 — Image with all the noise peaks removed



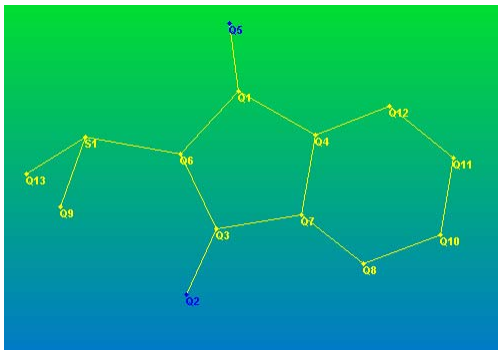
5. Put the cursor over the bonds to check distance. The distance is displayed at the bottom right.

8.3.2 Labelling the Atoms

1. Left-click the peaks for the two oxygen atoms to select them (Figure 8.14).

NOTE: If it is difficult to see the color and labels, change the color scheme with **Preferences > Background Color**. Choose colors and click **Apply**. Click **Cancel** to exit the background color mode.

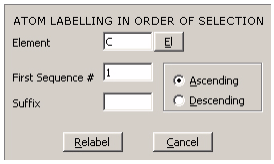
Figure 8.14 —Model with probable oxygen peaks selected



2. Right-click in the display, and choose **Labelling...** from the right-click menu.

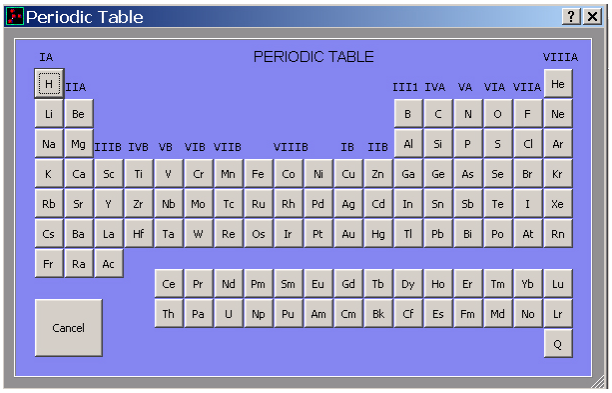
   The Atom Labelling box opens.

Figure 8.15 —Atom labelling box



3. Label the selected atoms as oxygen. Do this in one of two ways:
   - Click the “Element” field and type the element symbol (case does not matter).
   - Click the **El** button to the right of the “Element” field to open a periodic table. Click the appropriate element symbol to choose it (the periodic table will automatically close).

Figure 8.16 —Periodic table for choosing atom types



4. Click **Relabel** to relabel and number the peaks sequentially in the order they were selected, starting with the number in the “First Sequence #” field.
5. To label the carbon atoms, select the remaining peaks in the order you want them labeled.
6. In the labelling tool, change the element type to C (carbon).

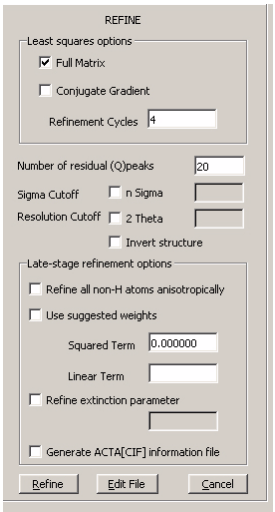
   The starting atom number changes to 1 (i.e., the first available number for carbon atoms).
7. Click **Relabel** to relabel the carbon atoms.
8. When all atoms are labeled and the molecule is ready for refinement, click **Cancel**.

8.3.3 Refining the Model

1. In XSHELL’s Tool Icon Bar, click the **Refine** icon , press [Ctrl]+[R], or choose **Refine > Refine**.

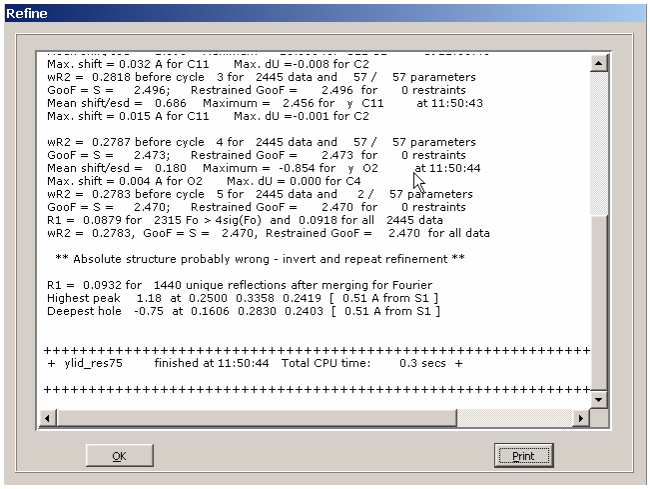
   The “Refine” window opens.
2. Click **Refine** to launch XL (the least-squares refinement program) using the default parameters as given in Figure 8.17.

Figure 8.17 —“Refine” menu



After a few seconds, the “Refine” window opens containing output results for the calculation.

Figure 8.18 —Isotropic refinement output

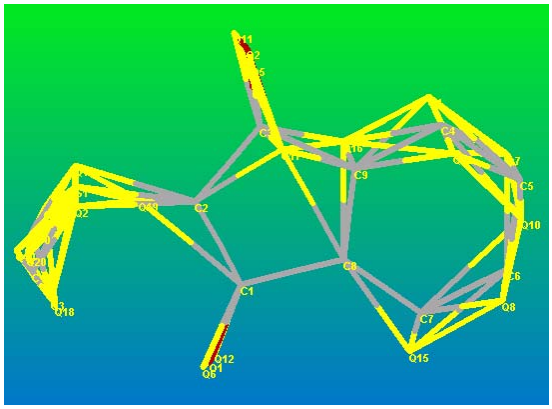


In the output displayed above, note that the R1 value is 0.09. This is typical for a preliminary isotropic refinement for an organic molecule with data to a resolution of 0.75Å, and no hydrogen atoms included. The refinement program also indicates that the model needs to be inverted to get the correct absolute structure.

NOTE: This indication of the absolute configuration is usually reliable, but all assignments of absolute structure should be confirmed later in the refinement process by including `TWIN` and `BASF` commands in the instruction input.

3. Click **OK** to return to XSHELL. The display refreshes with the results of the least-squares calculations.

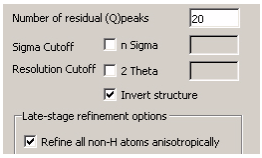
Figure 8.19 —Model after isotropic refinement



The peaks in the diagram represent differences in the electron density between the refinement model and the experimental electron density as defined by the measured data. Many of these difference peaks are near the sulfur and oxygen atoms. Refinement of all atoms anisotropically should improve the model.

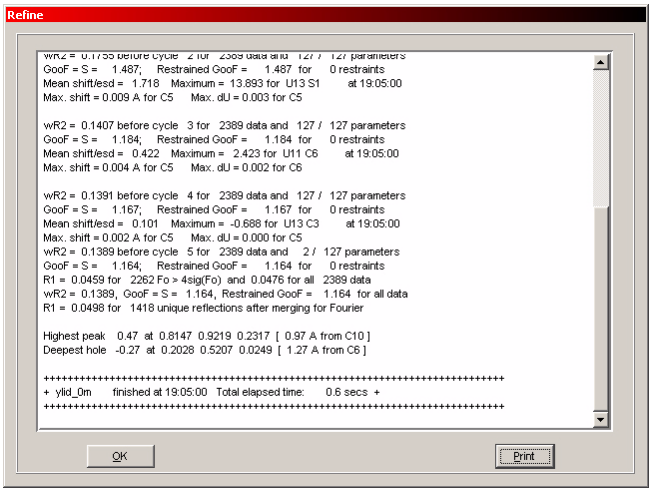
4. In the “Refine” menu, activate the **Invert Structure** and **Refine All Non-H Atoms Anisotropically** checkboxes as in Figure 9.24.

Figure 8.20 —Inverting and choosing anisotropic refinement



5. Click **Refine** to launch the least-squares refinement program. The output window opens and a summary of the results of individual cycles of refinement appears.

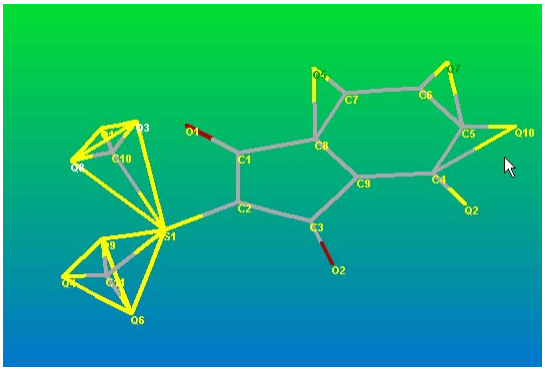
Figure 8.21 —Least-squares refinement summary



The value for R1, 0.0459, is typical for an initial anisotropic refinement with no hydrogen atoms included. Since the mean shift/esd is greater than 0.1, the model is still changing. Since the highest peak in the difference map has a height of 0.47 and is 0.97Å from C10, there are certainly some hydrogen atoms to be included.

Examination of the difference peaks in the resulting difference electron density map shows that the top difference peaks all correspond to hydrogen atoms.

Figure 8.22 —Difference peaks correspond to expected hydrogen atom positions

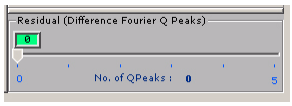


Viewing Atomic Displacement Parameters

It is generally good practice to examine the displacement parameters at this point in structure determination.

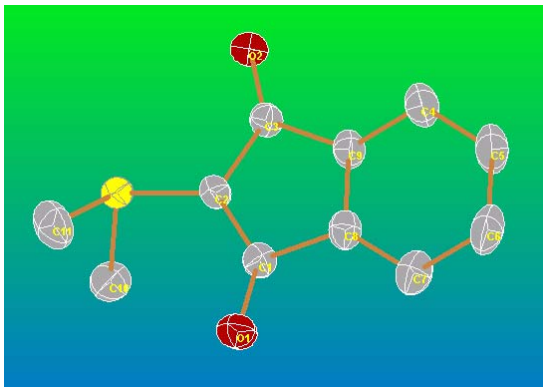
1. Remove difference peaks with the slider tool.

Figure 8.23 —Removing all Q peaks with the slider bar



2. Right-click in the background, and choose **Thermal Ellipsoids**.

Figure 8.24 —Thermal ellipsoid plot



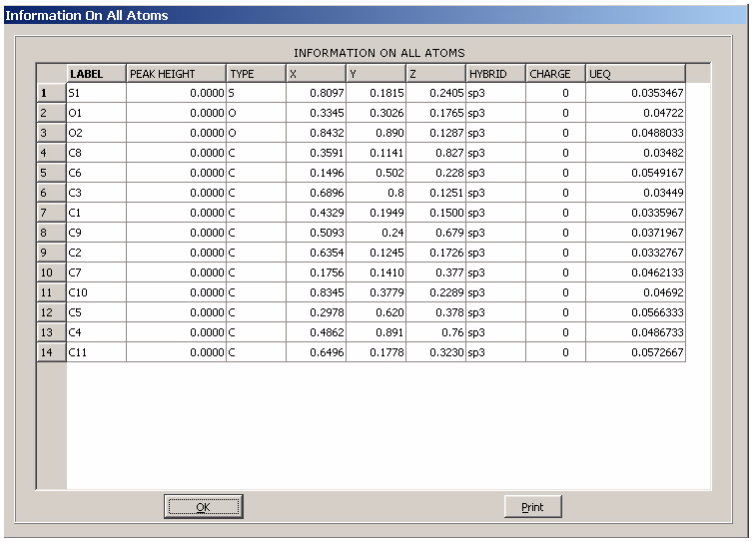
Generating a List of Equivalent Isotropic Displacement Parameters

In addition to the thermal ellipsoid plot, a list of Ueqs (equivalent isotropic displacement parameters) can be generated by requesting information on all of the atoms. Examining these values for outliers is good practice.

1. Right-click in the background, and choose **Information on All Atoms**.

   The “Information on All Atoms” window appears.

Figure 8.25 —List of parameters with Ueq to the right



	LABEL	PEAK HEIGHT	TYPE	X	Y	Z	HYBRID	CHARGE	UEQ
1	S1	0.0000	S	0.8097	0.1815	0.2405	sp3	0	0.0353467
2	O1	0.0000	O	0.3345	0.3026	0.1765	sp3	0	0.04722
3	O2	0.0000	O	0.8432	0.890	0.1287	sp3	0	0.0488033
4	C8	0.0000	C	0.3591	0.1141	0.827	sp3	0	0.03482
5	C6	0.0000	C	0.1496	0.502	0.228	sp3	0	0.0549167
6	C3	0.0000	C	0.6896	0.8	0.1251	sp3	0	0.03449
7	C1	0.0000	C	0.4329	0.1949	0.1500	sp3	0	0.0335967
8	C9	0.0000	C	0.5093	0.24	0.679	sp3	0	0.0371967
9	C2	0.0000	C	0.6354	0.1245	0.1726	sp3	0	0.0332767
10	C7	0.0000	C	0.1756	0.1410	0.377	sp3	0	0.0462133
11	C10	0.0000	C	0.8345	0.3779	0.2289	sp3	0	0.04692
12	C5	0.0000	C	0.2978	0.620	0.378	sp3	0	0.0566333
13	C4	0.0000	C	0.4862	0.891	0.76	sp3	0	0.0486733
14	C11	0.0000	C	0.6496	0.1778	0.3230	sp3	0	0.0572667

2. Sort the atoms.

Reasonable Ueq Values

For the ylid molecule, the Ueq values are reasonable if there are:

- lower values for the sulfur atom and the five-member ring;
- higher values for the terminal atoms — methyls and carbonyls; and
- sensible numbers for the six-member ring — lower for C8 and C9, higher for C5 and C6, and in between for C4 and C7.

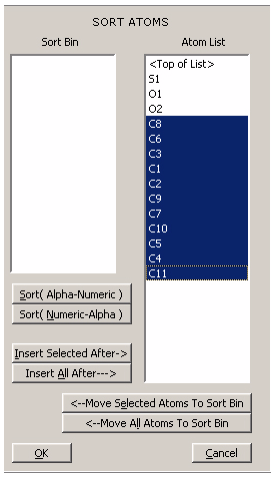
Sorting Atoms

When examining the values in the “Information on All Atoms” window is difficult because the atoms are not in order, the atoms may be sorted.

1. Right-click on the window’s background, and choose **Sort…** to sort the atoms into a sensible order.

   An Atom List box appears to the right of the molecule display. Since S1, O1 and O2 are in a reasonable order, only the carbon atoms need to be ordered.

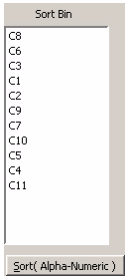
Figure 8.26 —Selecting carbons for sorting



NOTE: Generally, it is easier to sort and number atoms before adding hydrogen atoms.

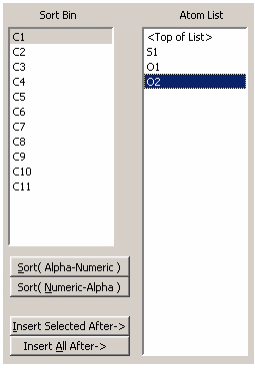
2. Select the atoms to be sorted (i.e., click and drag over the atoms) and move them to the Sort Bin (i.e., click **Move Selected Atoms to Sort Bin**).

Figure 8.27 —carbon atoms in Sort Bin



3. After the atoms are in the Sort Bin, click **Sort (Alpha-Numeric)**.
4. Select “O2”, and click **Insert All After** as in .

Figure 8.28 —Sorted atoms with the “insert after” atom (i.e., O2) selected



5. Click **OK** to accept the sorting.

Adding Hydrogen Atoms

After refining the non-H-atoms, refine the hydrogen atoms either by:

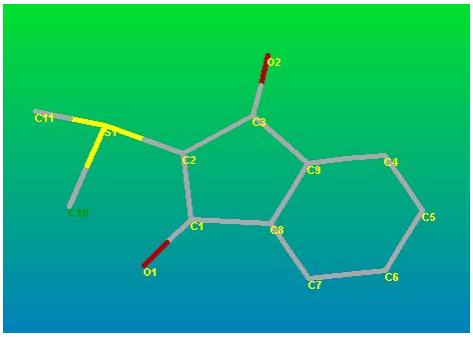
- renaming the Q-peaks using the Labelling tool, and then including them in the refinement; or
- including the hydrogen atoms in fixed idealized positions, riding on the atoms to which they are attached.

In this example, we illustrate the latter fixed idealized method.

NOTE: In some cases (e.g., hydrogen atoms on hetero atoms or on geometrically strained carbon atoms), it is better to refine the hydrogen atom positions. A general rule is to refine if there is some doubt about the position of the hydrogen atom and there is difference electron density present.

1. Use the slider tool to remove all of the Q peaks.

Figure 8.29 —Refined model with difference peaks removed



2. Right-click on the background, and choose **Add Hydrogen atoms > Hybridize All**.

   The colors of the atom labels change to indicate atom hybridization.

NOTE: If the atom hybridizations are hard to see, right-click on the background and open the “Information on All Atoms” window.

3. Right-click on the background, and choose **Add Hydrogen atoms > Calculate Hydrogens**.
4. A warning appears indicating that the hydrogen atoms have been provisionally added and that the final addition with naming will take place when the least-squares calculations are started (Figure 8.30). Click **OK**.

Figure 8.30 —Hydrogen atom warning

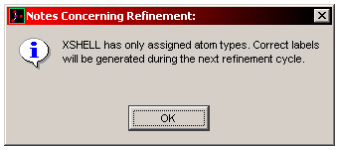
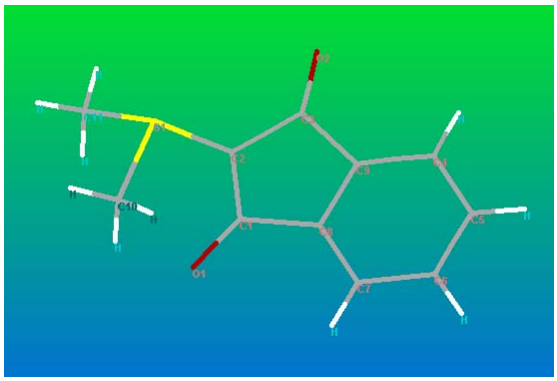


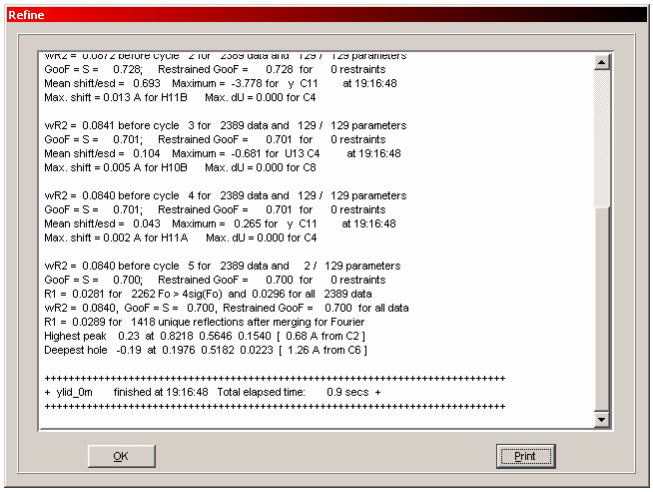
Figure 8.31 —Model with added hydrogen atoms



5. Click **Refine** to launch the least-squares refinement program.

   The output window opens, and a summary of the results of individual refinement cycles appears.

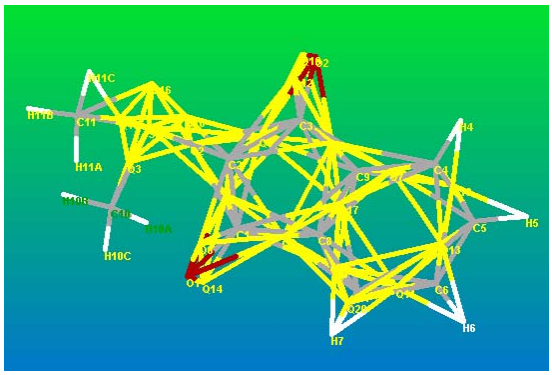
Figure 8.32 —Refinement results



The value for R1, 0.0281, is typical for an anisotropic refinement with riding hydrogen atoms and a data set measured to 0.75Å. Since the mean shift/esd is less than 0.01, the model is not changing significantly. Since the highest peak and the deepest hole in the difference map are similar, there are probably no missing atoms.

In the resulting difference electron density map shown in Figure 8.33, the number of difference peaks to be displayed has been left at 20.

Figure 8.33 —Difference peaks in a well-refined model



8.3.4 Refining for Publication with XSHELL

1. Obtain a view that is much easier to interpret by reducing the number of difference peaks in the “Refine” menu (Figure 8.34).
2. For the final cycles of refinement, reduce the number of peaks to be displayed to five and turn on “Use Suggested Weights” and “Generate ACTA(CIF) Information File” as shown in the next two figures.

NOTE: If the suggested weights vary significantly from 0.06 and 0.00, then change the values to 0.06 and 0.00.

Figure 8.34 —Reduce the number of difference peaks

Figure 8.35 —Select weights and request ACTA output

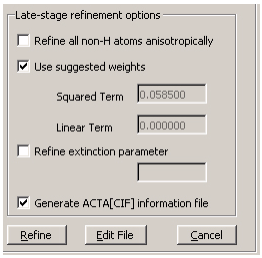
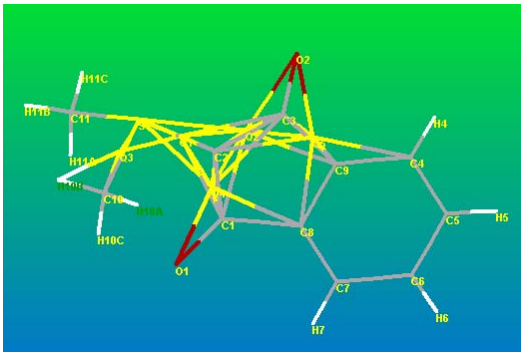


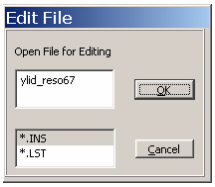
Figure 8.36 shows a difference map with the number of peaks set at 5.

Figure 8.36 —Difference peaks



3. Click the **Edit File** button, choose the .ins file, and click **OK** to open the instruction file for XL. This allows you to add instructions that are not directly available in the GUI.

Figure 8.37 —“Edit File” window



4. Add the desired additional commands.
    4.1. Check that the `TEMP` (i.e., temperature) command is correct.
    4.2. For torsion angles, add a `CONF` (i.e., confirmation) command.
    4.3. If planarity and angles between planes are of interest, add `MPLA` commands.

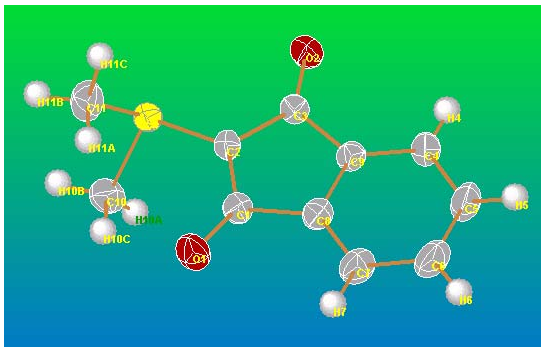
Figure 8.38 —Additional instructions in the .ins file

```
TEMP 23
conf
mpla 12 c1 c2 c3 c4 c5 c6 c7 c8 c9  s1  o1 o2 c10 c11
mpla 6 c4 c5 c6 c7 c8 c9 c1 c2 c3  o1 o2 s1 c10 c11
mpla 5 c8 c9 c1 c2 c3 c4 c5 c6 c7  o1 o2 s1 c10 c11
mpla 10 c1 c2 c3 c4 c5 c6 c7 c8 c9  s1  o1 o2 c10 c11
```

8.3.5 Creating a Publication-Quality Image

1. Remove difference peaks with the slider tool.
2. Right-click on the background, and choose **Thermal Ellipsoids**.

Figure 8.39 —Thermal ellipsoids with large hydrogen atoms



3. If the hydrogen atoms in Figure 8.39are large, change their sizes:
    3.1. Choose **Preferences > Atom Preferences...** in XSHELL's Menu Bar.
    3.2. In the "Atom Preferences" area, change the hydrogen radius to 0.08 and the bond radius to 0.15 to make the hydrogen atoms smaller. Click **Apply**.

Figure 8.40 —"Atom Preferences" menu

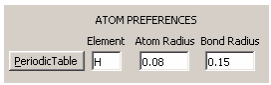
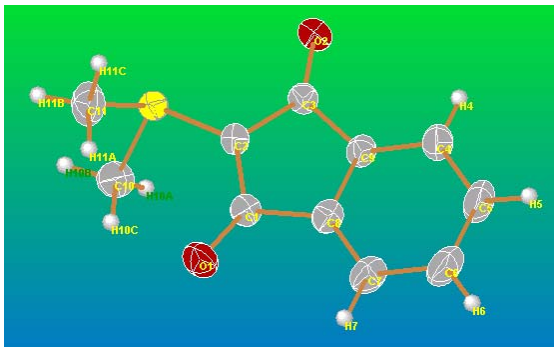
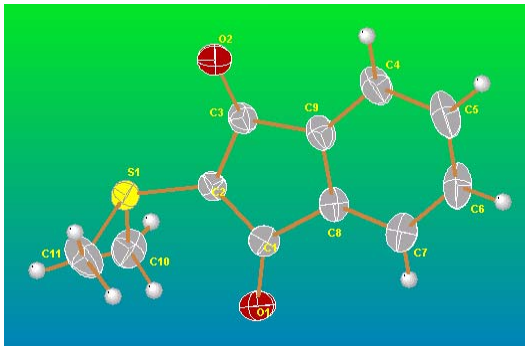


Figure 8.41 —Thermal ellipsoid plot with smaller hydrogen atoms



4. Position atom labels by right-clicking on the atom and choosing **Position Label**.

Figure 8.42 —Positioned atom labels



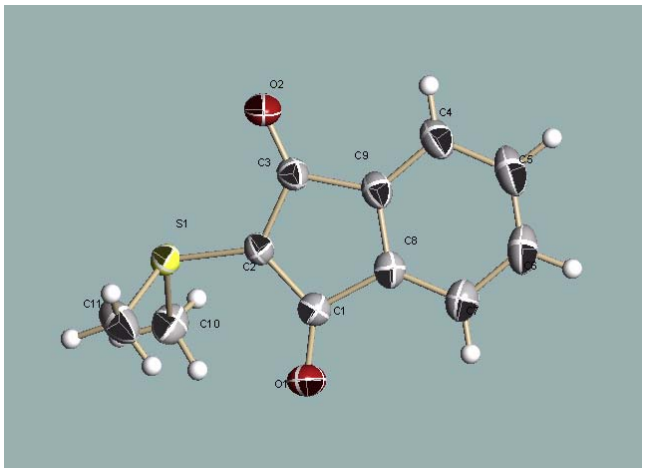
5. Convert this image into a high-quality .jpg file for inclusion into reports by choosing **Render > Color > To High Quality JPEG File**.

NOTE: A message may appear that the font file needs to be defined: “Please set your font directory for the first time for opengl rendering.” If this happens, choose **Preferences > OpenGL Preferences**, and click on the TrueType font For.OpenGL.

NOTE: In Windows, browse to C:\bn\xshell\fonts\

NOTE: In Linux, navigate to /usr/local/bin/xshell/fonts. Choose a font.

Figure 8.43 —Final rendered thermal ellipsoid/atomic displacement parameter plot



This page intentionally left blank.

9 Using the Report Generation Module

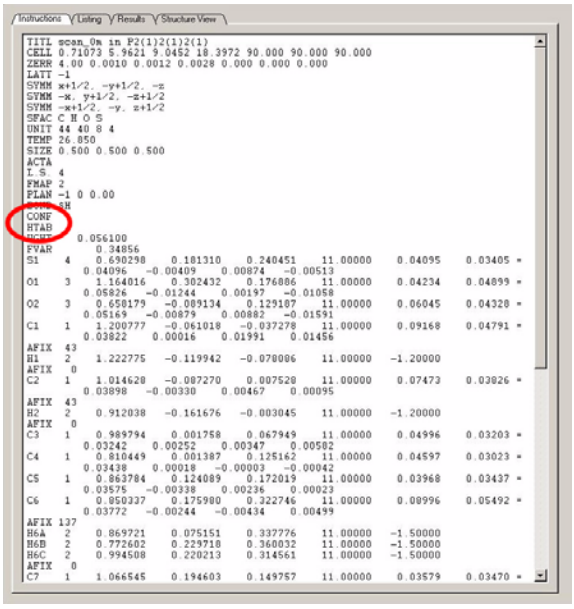
9.1 Preparing for Report Generation

Before creating the report, make sure that your *.ins file has the correct values for the final structure:

- Correct formula (`SFAC` and `UNIT` instructions)
- Correct Z value (`ZERR` instruction)
- Final cell constants with ESDs (from the final *_0m._ls file)
- Correct atom sorting and labelling
- `TEMP` instruction (in °C) (e.g., `TEMP -173` for 100K)
- `SIZE` instruction
- `ACTA` instruction
- `BOND $H` instruction
- `CONF` instruction for torsion angles (if applicable)
- `HTAB` instruction for hydrogen bonds (if applicable)

NOTE: You may need to edit the instructions file to add one or more of these instructions or parameters. If so, click **Refine with XL** to generate the .cif file that you will import into the report.

Figure 9.1 — Instructions added to .ins file

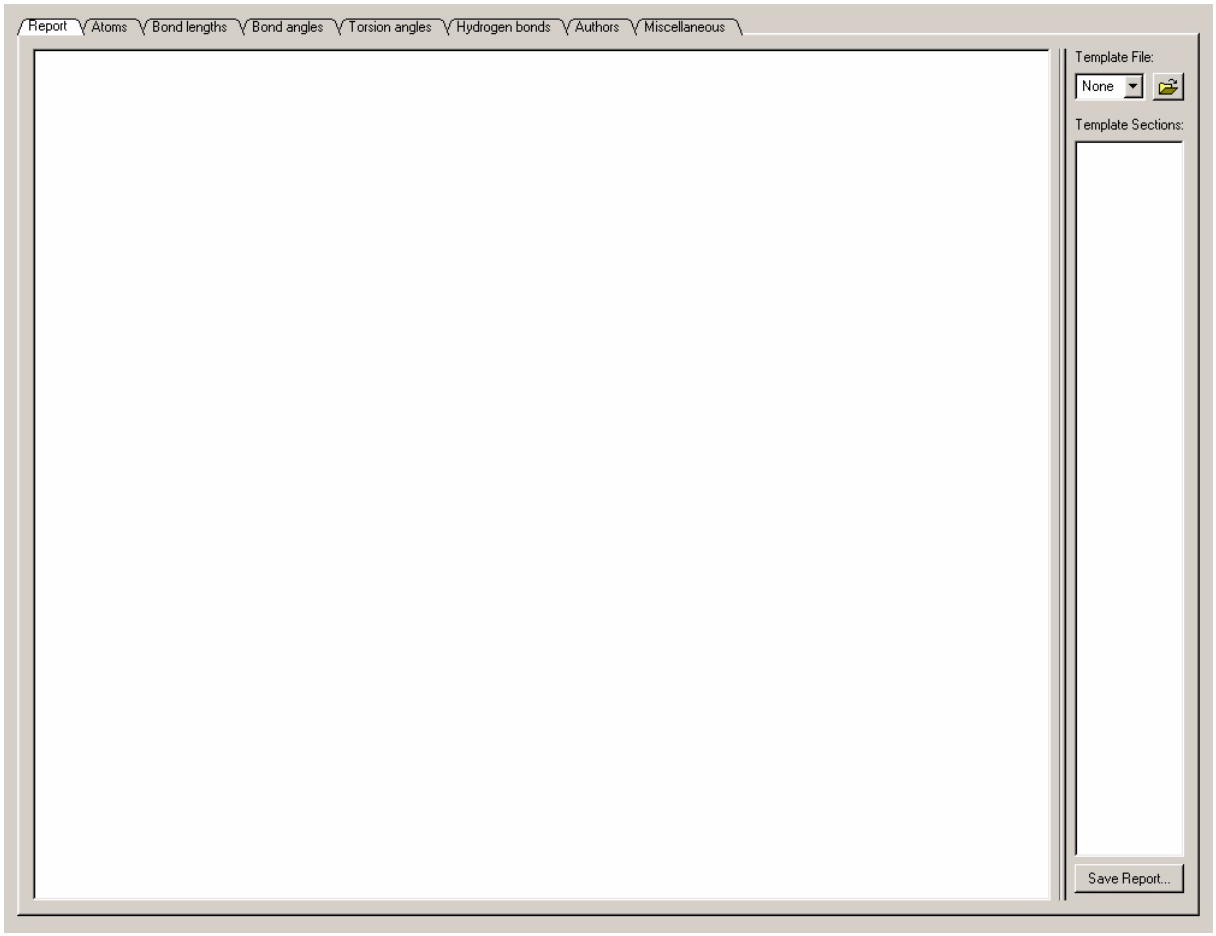


9.2 Using the Module

9.2.1 Starting the Module

1. In the Task Bar's "Report" category, click the **Generate Report** icon .
   The Generate Report module opens.

Figure 9.2 — Generate Report module: initial view

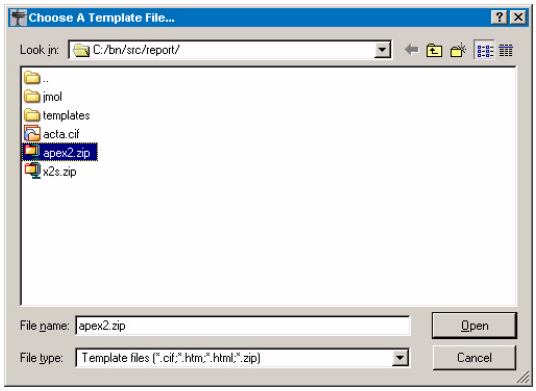


9.2.2 Choosing a Template File

To set the formatting for your report, you will choose a template on which your report will be based. Template files are contained in the directory C:/bn/src/report.

1. In the right-hand section of the module, use the drop-down menu or the **Browse** button to find the desired template file:
   - Choose apex2.zip to format the report as output from the module, without instrument-specific text.
   - Choose x2s.zip to format the report with SMART X2S-specific text and data.
   - Choose acta.cif to format the report as a .cif file for submission to *Acta Crystallographica*.

Figure 9.3 — Choosing a template file



2. Click **Open**.

   The module retrieves your project's data and populates the report based on the selected template.

Proceed to Section 9.3 for information on creating a report for online or print distribution.

Proceed to Section 9.4 for information on creating a report for submission to *Acta Crystallographica*.

9.3 Creating an HTML-Formatted Report for Online or Print Distribution

Bruker's report format is an HTML file, along with necessary Java files to display the structure using the free structure viewer Jmol. The report may be viewed on any computer with Java and a web browser. The HTML file may also be opened in Microsoft Word or other word-processing programs to produce printed manuscripts.

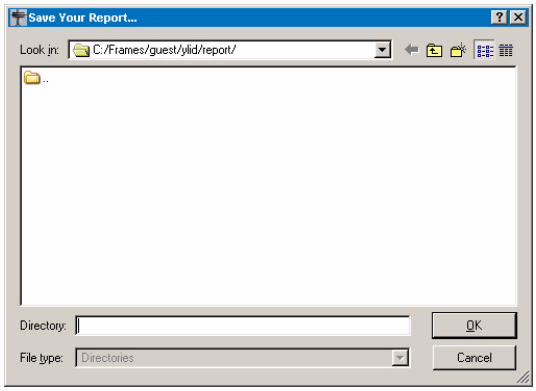
Before saving the report, add any additional information (e.g., information from your .cif file, your run lists, your contact information) to the report.

9.3.1 Saving the HTML-Formatted Report

1. Under the "Report" tab, click the **Save Report...** button.

   The "Save Your Report..." dialog opens.

Figure 9.4 — "Save Your Report..." dialog



2. Choose the desired directory for your report (there is no file name to type in; the final report will be named report.html, located in the chosen folder with the other required files).
3. Click **OK**.

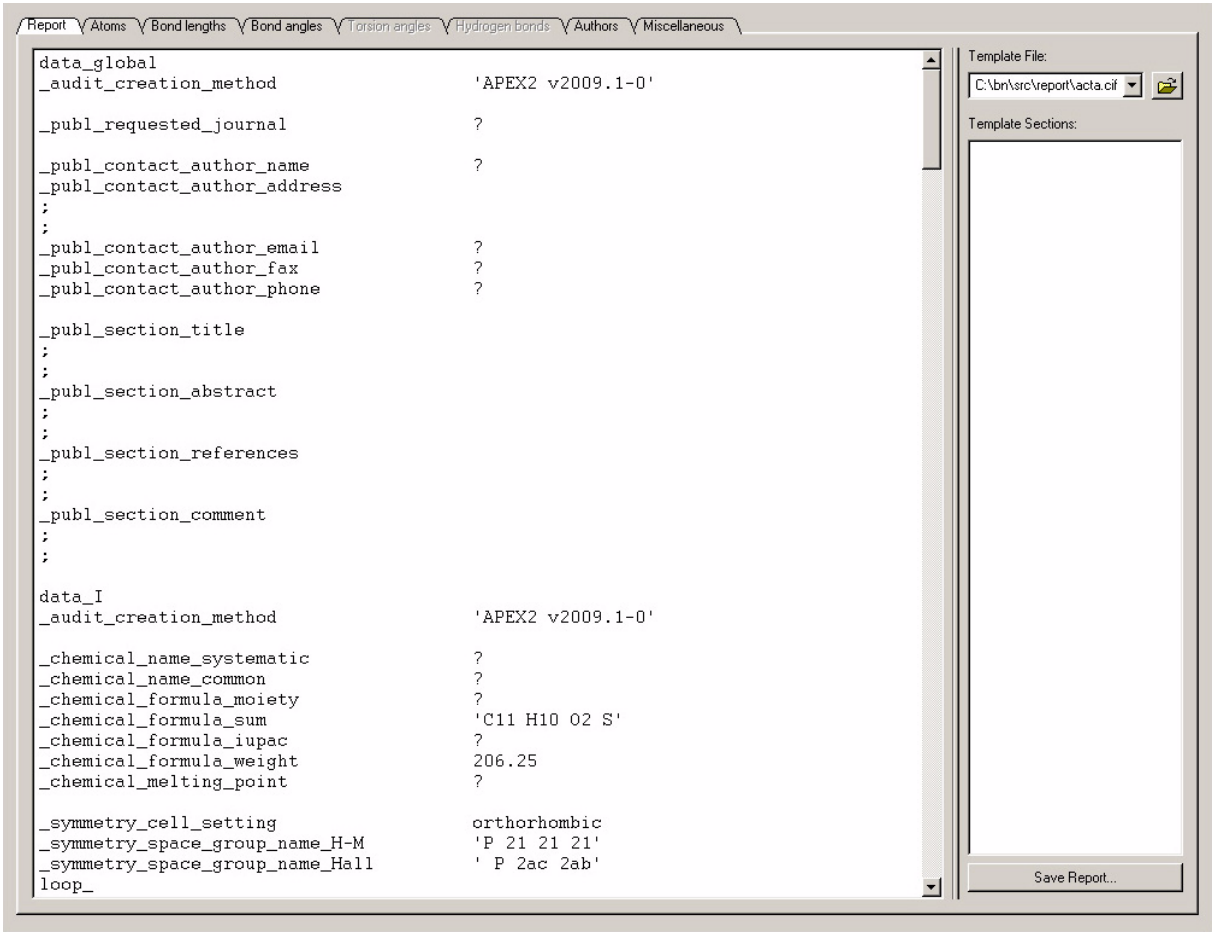
   The report and additional files are saved to the specified directory.

9.4 Creating a Report for Submission to *Acta Crystallographica*

Before saving the report, add any additional information (e.g., information from your .cif file, your run lists, your contact information) to the report.

NOTE: If you need information from the .cif file generated by XL, you will need to import it regardless of whether you have already created an HTML-formatted report.

Figure 9.5 — CIF report initial view



9.4.1 Saving the CIF-Formatted Report

1. Under the “Report” tab, click **Save Report...**.

   The “Save” dialog opens.

2. Choose the desired directory for your report.

3. Click **Save**.

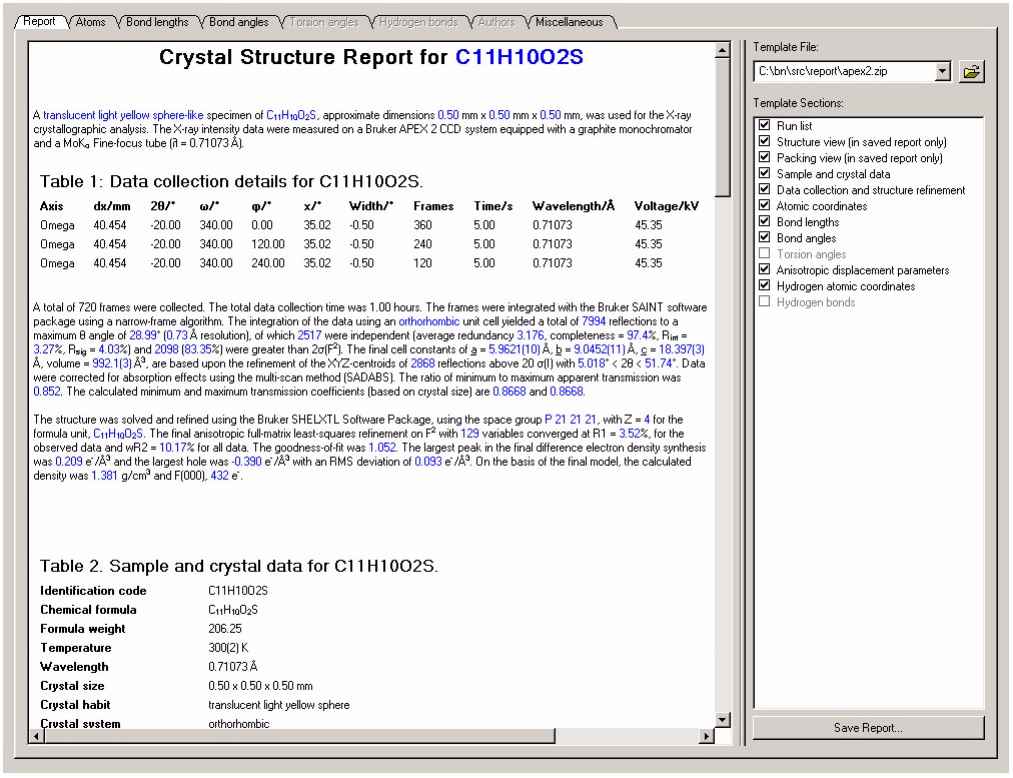
   The report is saved to the specified directory.

9.5 Adding Additional Information to the Report

To achieve a publication-quality report, you will need to add certain information to the report before saving it. This information may include:

- Information from the .cif file generated by XL (e.g., torsion angles, hydrogen bonds)
- The experiment's run list
- Additional information (e.g., diffractometer information, name of institution, contact information, etc.)

Figure 9.6 — Torsion and hydrogen bonds missing



9.5.1 Adding Information from a .CIF File

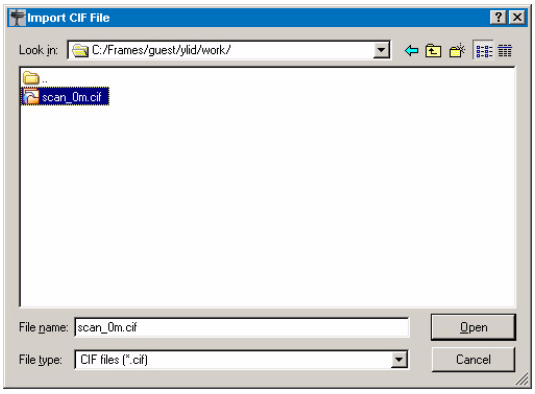
To add information from a .cif file:

1. Choose **Report > Import XL CIF File...** from the Menu Bar.

   The “Import CIF File” window opens.

Figure 9.7 — **Report > Import XL CIF File...**

Figure 9.8 — “Import CIF File” dialog



2. Browse to the desired .cif file, and click **Open**.

   The .cif file’s information is imported into the report. If information is already present in the imported fields, it is overwritten.

9.5.2 Adding the Run List

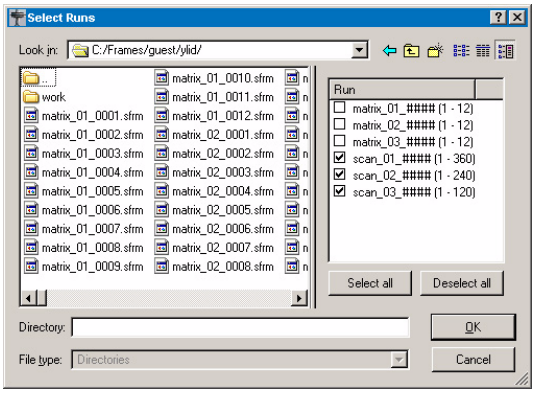
To add the run list:

1. Choose **Report > Import Run List...** from the Menu Bar.

   A run list selection dialog appears.

Figure 9.9 — **Report > Import Run List...**

Figure 9.10 —"Select Runs" dialog



2. Browse to your project's location, choose the desired runs, and click **OK**.

   The run list is imported into the report.

9.5.3 Adding Additional Information

To add additional information to the report, simply choose the desired tab (e.g., the “Miscellaneous” tab), and edit the information directly in the necessary fields. When you return to the “Report” tab, the new information will be updated in the report.

Importing and Exporting Additional Information

The “Miscellaneous” tab allows you to import and export report information (e.g., for generic information that is reused often, such as experiment setup and contact information).

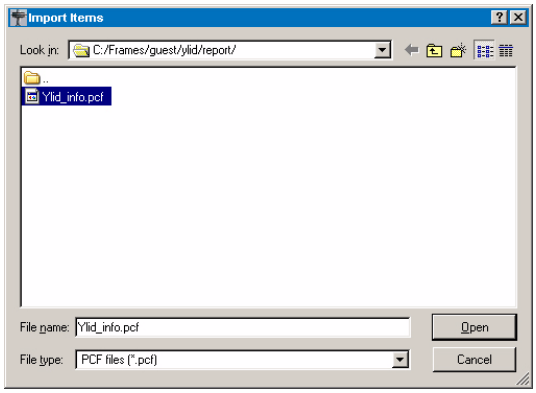
Importing Information

To import information into the “Miscellaneous” tab:

1. Click the **Import Items...** button.

   The “Import Items” dialog opens.

Figure 9.11 — “Import Items” dialog



2. Browse to the .pcf file that contains the desired information.
3. Click **Open**.

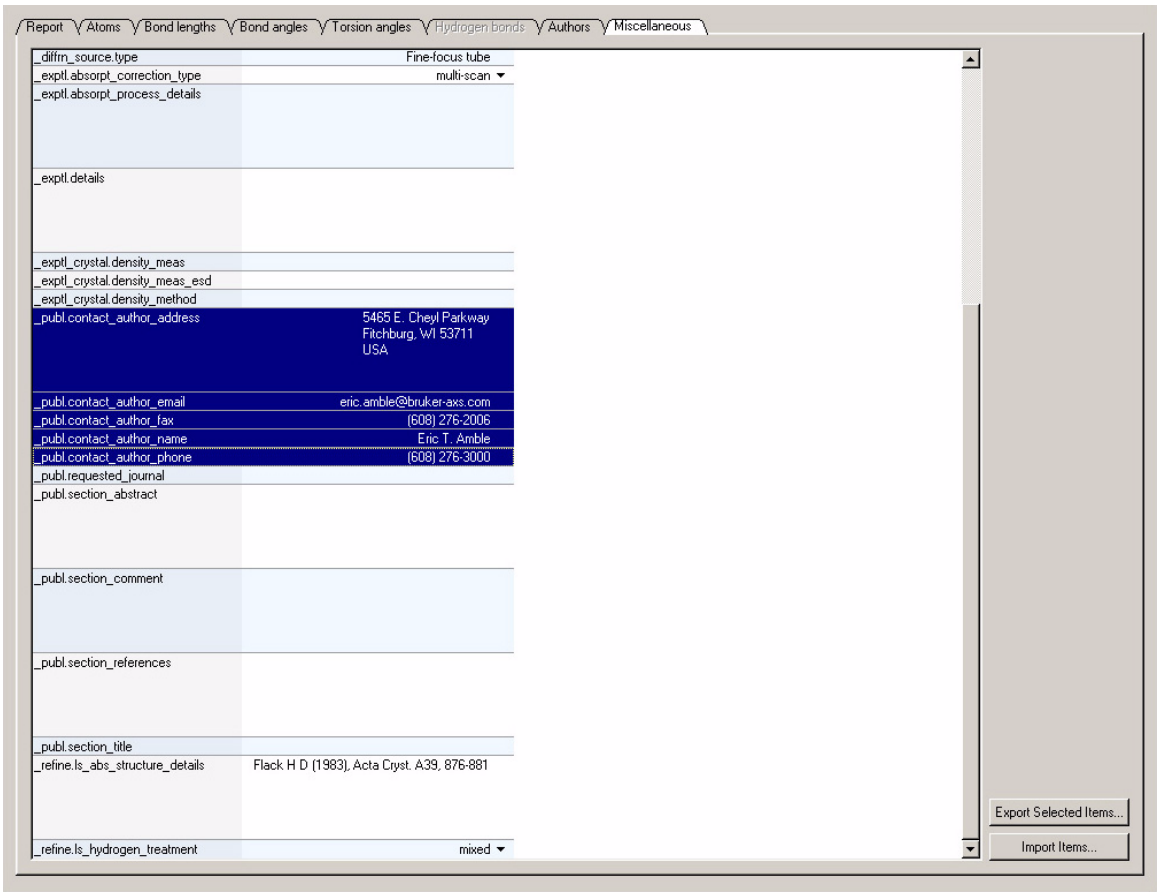
   The information is imported into the report. If information is already present in the imported fields, it is overwritten.

Exporting Information

To export information from the “Miscellaneous” tab:

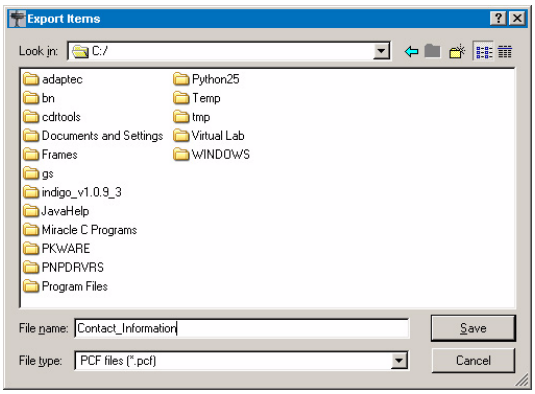
1. Click on the fields to select them. [Ctrl]-click to select multiple items, or [Shift]-click to select a range.

Figure 9.12 —Selecting data for export



2. Click the **Export Selected Items...** button.
   The “Export Items” dialog opens.

Figure 9.13 —“Export Items” dialog



3. Choose a directory and filename for the exported information, and click **Save** to save the information as a .pcf file.

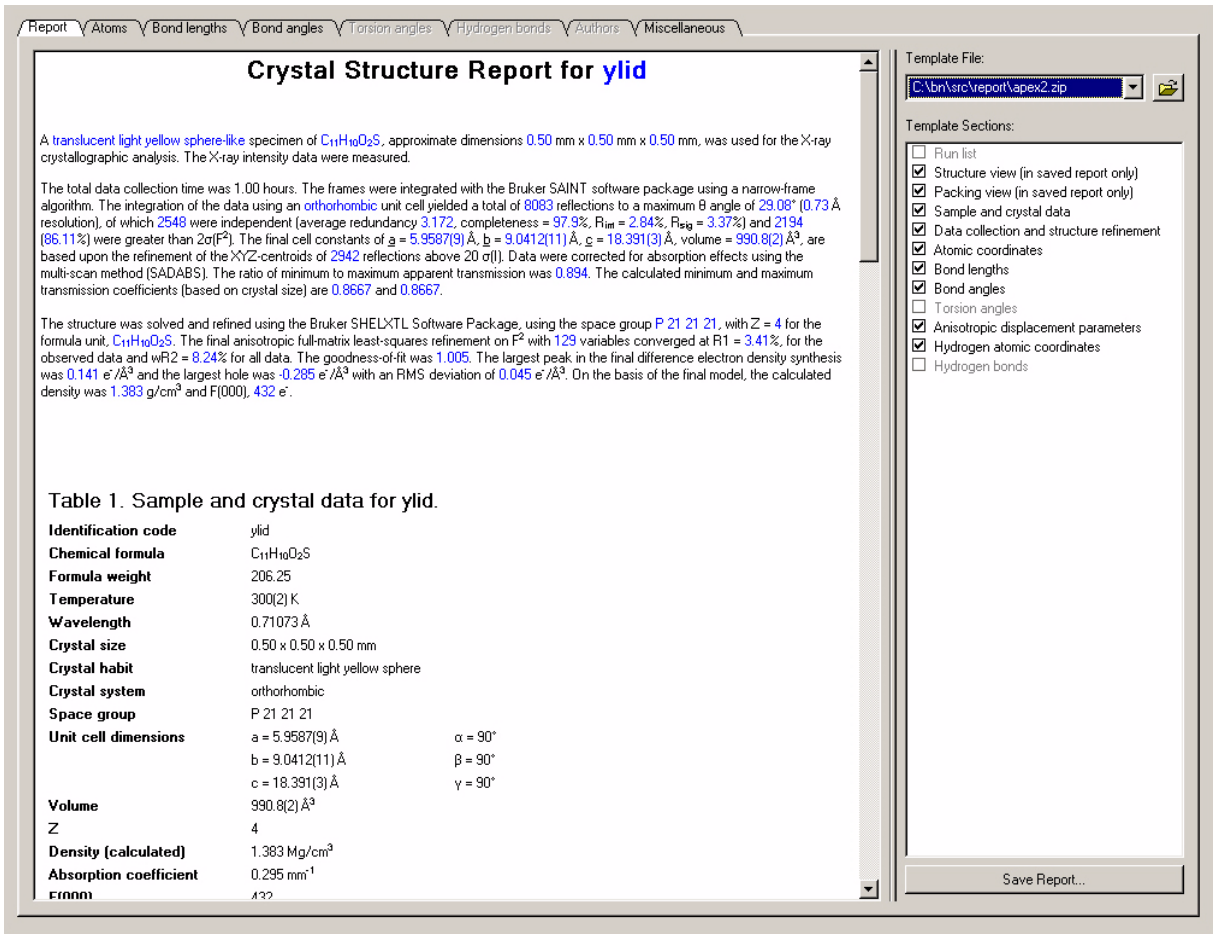
9.6 Report Sections

The module displays the report in a series of tabbed sections for review before saving.

9.6.1 "Report" Tab

The Report tab gives an at-a-glance view of the report as it will appear after saving (either as HTML or as a .cif file). It contains all of the sections that are shown in the other tabs.

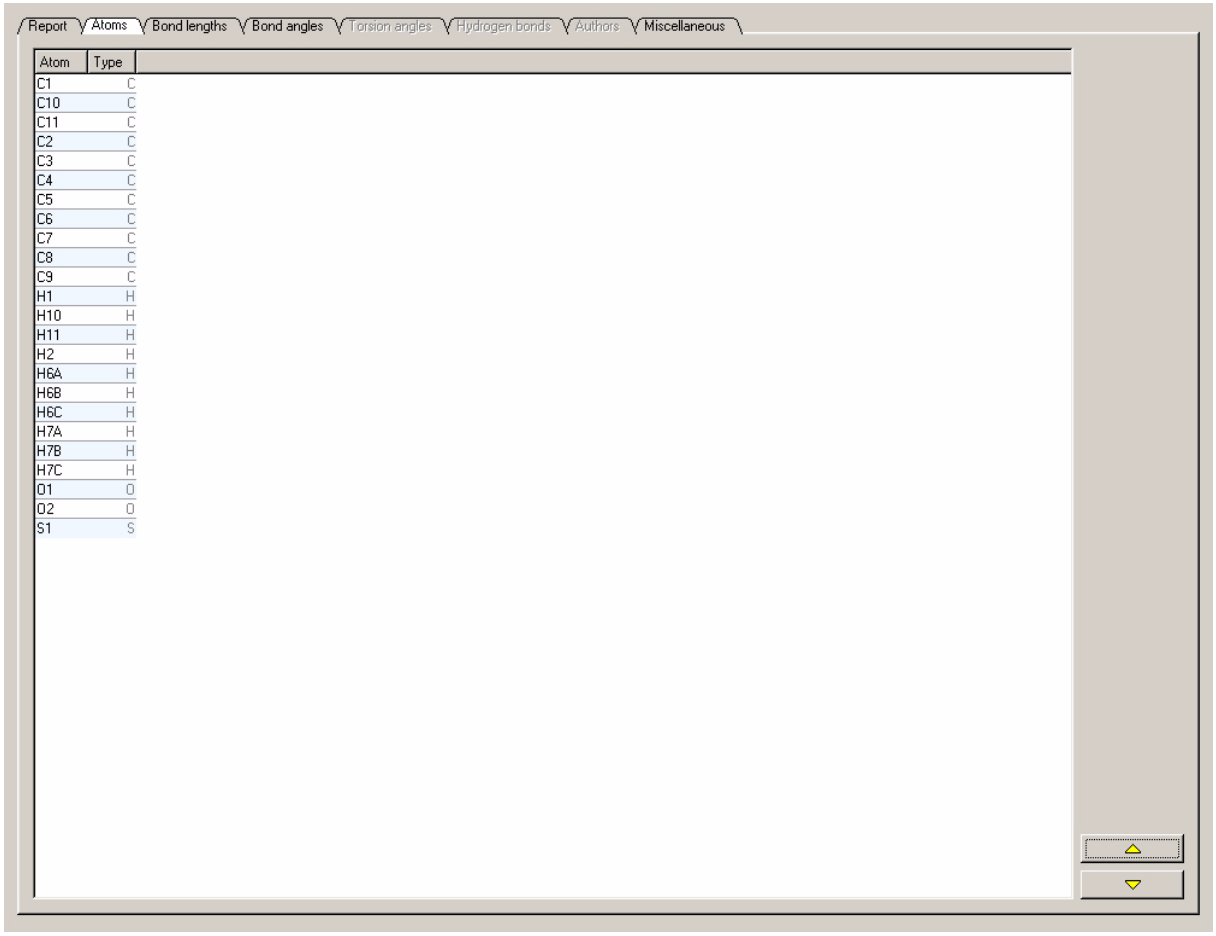
Figure 9.14 —"Report" tab



9.6.2 “Atoms” Tab

The “Atoms” tab lists the atoms in the solved structure by type. The columns are sortable.

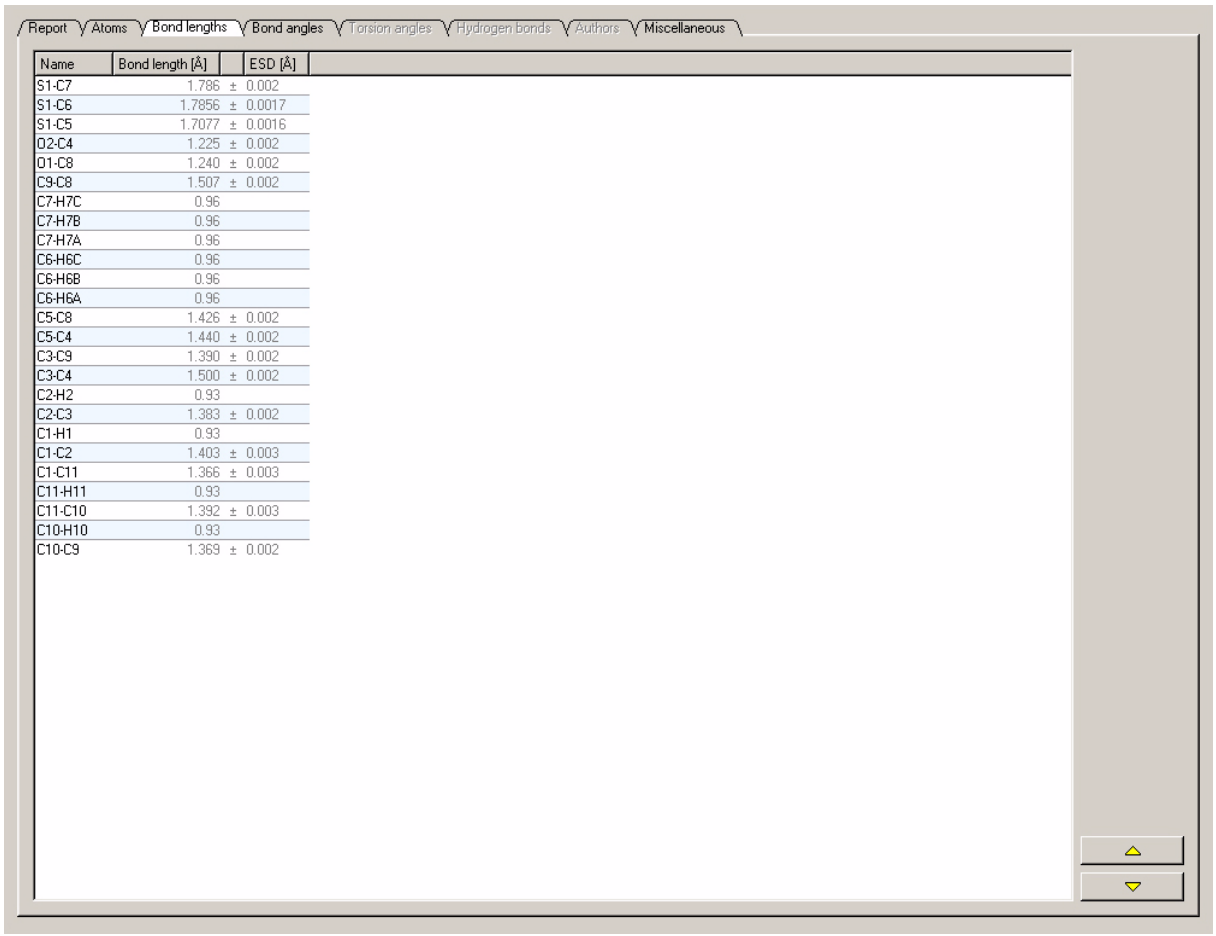
Figure 9.15 —“Atoms” tab



9.6.3 “Bond Lengths” Tab

Bond lengths and ESDs for the structure are given in this tab. The columns are sortable. Additionally, you can move an individual entry in the table up or down by selecting it with the mouse and clicking the and buttons in the lower right-hand corner.

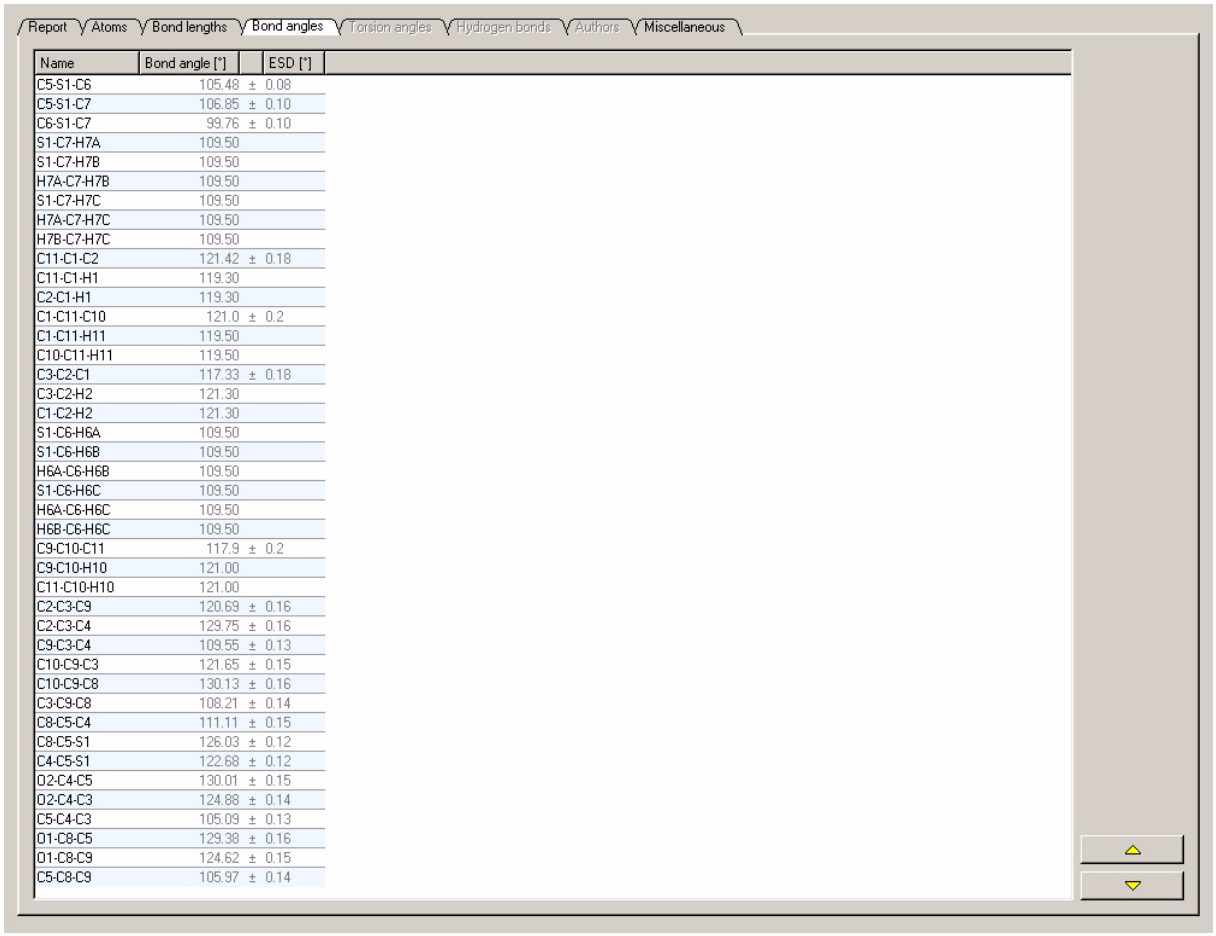
Figure 9.16 —“Bond Lengths” tab



9.6.4 Bond Angles

Bond angles and ESDs are given in this tab. The columns are sortable. Additionally, you can move an individual entry in the table up or down by selecting it with the mouse and clicking the ▲ and ▼ buttons in the lower right-hand corner.

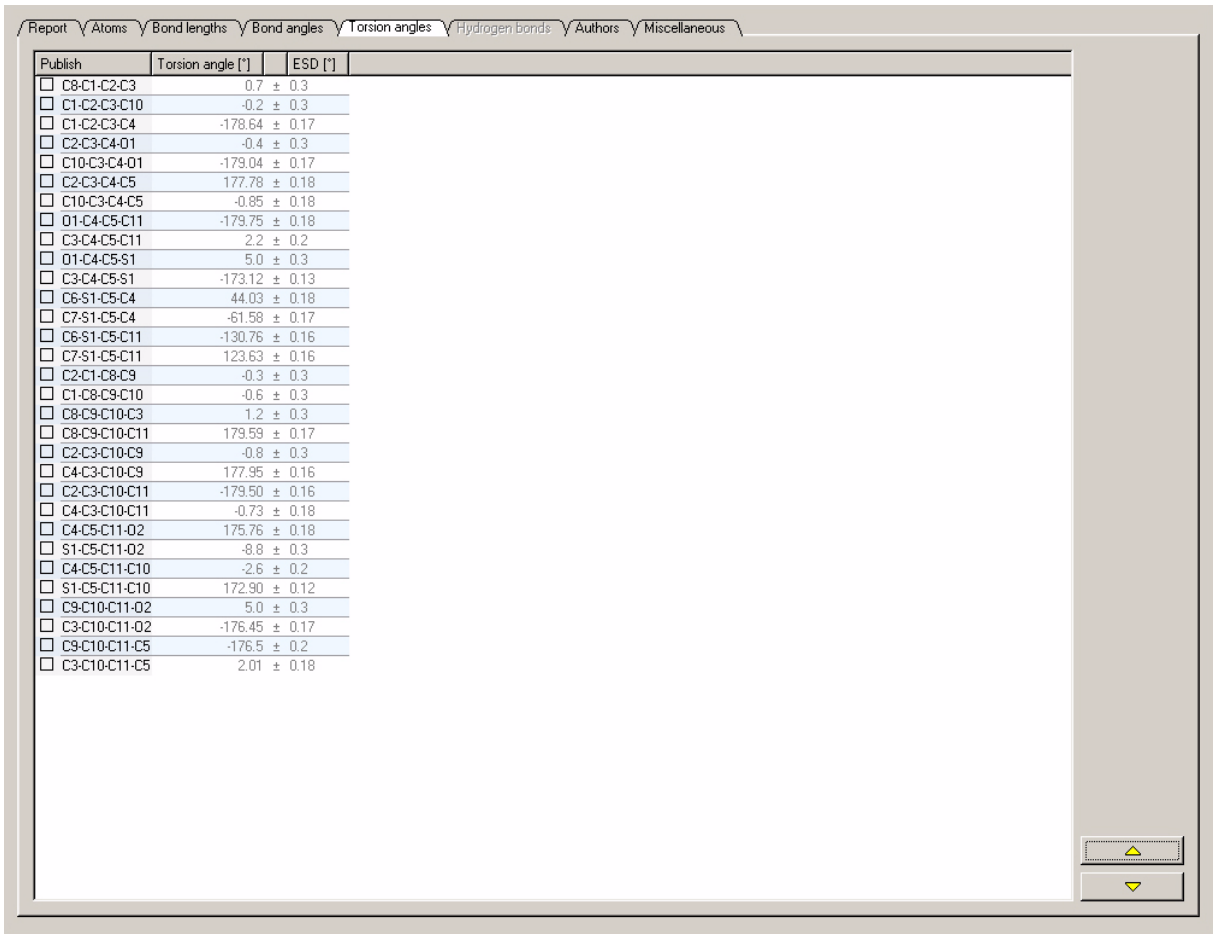
Figure 9.17 —"Bond Angles" tab



Name	Bond angle [°]		ESD [°]
C5-S1-C6	105.48	±	0.08
C5-S1-C7	106.85	±	0.10
C6-S1-C7	99.76	±	0.10
S1-C7-H7A	109.50		
S1-C7-H7B	109.50		
H7A-C7-H7B	109.50		
S1-C7-H7C	109.50		
H7A-C7-H7C	109.50		
H7B-C7-H7C	109.50		
C11-C1-C2	121.42	±	0.18
C11-C1-H1	119.30		
C2-C1-H1	119.30		
C1-C11-C10	121.0	±	0.2
C1-C11-H11	119.50		
C10-C11-H11	119.50		
C3-C2-C1	117.33	±	0.18
C3-C2-H2	121.30		
C1-C2-H2	121.30		
S1-C6-H6A	109.50		
S1-C6-H6B	109.50		
H6A-C6-H6B	109.50		
S1-C6-H6C	109.50		
H6A-C6-H6C	109.50		
H6B-C6-H6C	109.50		
C9-C10-C11	117.9	±	0.2
C9-C10-H10	121.00		
C11-C10-H10	121.00		
C2-C3-C9	120.69	±	0.16
C2-C3-C4	129.75	±	0.16
C9-C3-C4	109.55	±	0.13
C10-C9-C3	121.65	±	0.15
C10-C9-C8	130.13	±	0.16
C3-C9-C8	108.21	±	0.14
C8-C5-C4	111.11	±	0.15
C8-C5-S1	126.03	±	0.12
C4-C5-S1	122.68	±	0.12
O2-C4-C5	130.01	±	0.15
O2-C4-C3	124.88	±	0.14
C5-C4-C3	105.09	±	0.13
O1-C8-C5	129.38	±	0.16
O1-C8-C9	124.62	±	0.15
C5-C8-C9	105.97	±	0.14

9.6.5 “Torsion Angles” Tab

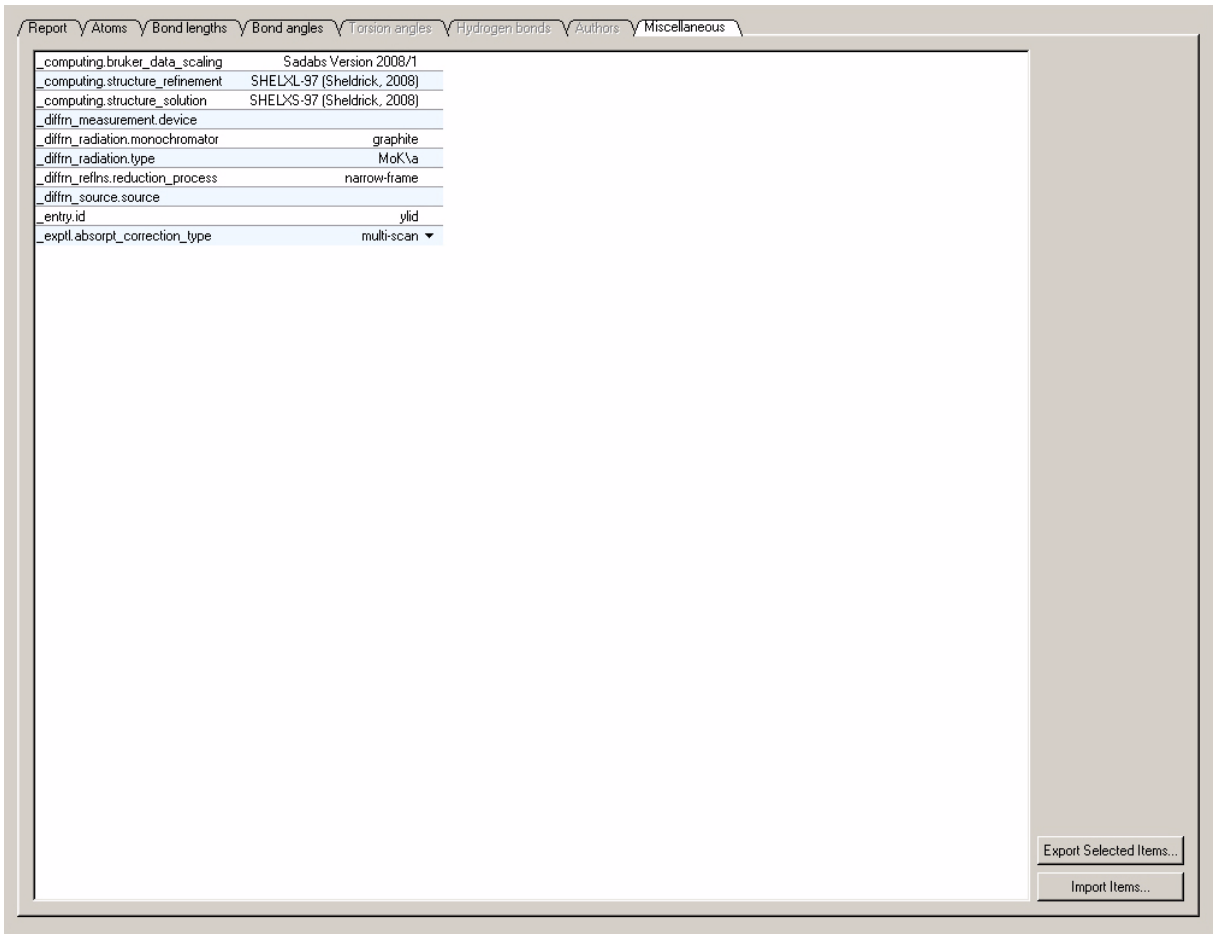
Figure 9.18 —“Torsion Angles” tab



Publish	Torsion angle [°]		ESD [°]
C8-C1-C2-C3	0.7	±	0.3
C1-C2-C3-C10	-0.2	±	0.3
C1-C2-C3-C4	-178.64	±	0.17
C2-C3-C4-O1	-0.4	±	0.3
C10-C3-C4-O1	-179.04	±	0.17
C2-C3-C4-C5	177.78	±	0.18
C10-C3-C4-C5	-0.85	±	0.18
O1-C4-C5-C11	-179.75	±	0.18
C3-C4-C5-C11	2.2	±	0.2
O1-C4-C5-S1	5.0	±	0.3
C3-C4-C5-S1	-173.12	±	0.13
C6-S1-C5-C4	44.03	±	0.18
C7-S1-C5-C4	-61.58	±	0.17
C6-S1-C5-C11	-130.76	±	0.16
C7-S1-C5-C11	123.63	±	0.16
C2-C1-C8-C9	-0.3	±	0.3
C1-C8-C9-C10	-0.6	±	0.3
C8-C9-C10-C3	1.2	±	0.3
C8-C9-C10-C11	179.59	±	0.17
C2-C3-C10-C9	-0.8	±	0.3
C4-C3-C10-C9	177.95	±	0.16
C2-C3-C10-C11	-179.50	±	0.16
C4-C3-C10-C11	-0.73	±	0.18
C4-C5-C11-O2	175.76	±	0.18
S1-C5-C11-O2	-8.8	±	0.3
C4-C5-C11-C10	-2.6	±	0.2
S1-C5-C11-C10	172.90	±	0.12
C9-C10-C11-O2	5.0	±	0.3
C3-C10-C11-O2	-176.45	±	0.17
C9-C10-C11-C5	-176.5	±	0.2
C3-C10-C11-C5	2.01	±	0.18

9.6.6 “Miscellaneous” Tab

Figure 9.19 —“Miscellaneous” tab



10 Appendices

10.1 Using BCP with APEX2

10.1.1 Start BCP

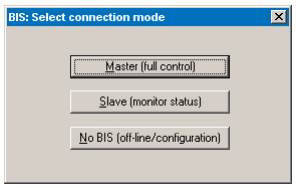
1. Choose **Start > Programs > Bruker AXS > Administration > BCP**, or double-click the desktop icon.

   The BCP main window opens.

10.1.2 Select a Connection Mode

When BCP starts, it prompts you to choose a connection mode.

Figure 10.1 —"Select connection mode" window



1. Click the appropriate button:
   - **Master (full control)**
   - **Slave (monitor status)**
   - **No BIS (off-line/configuration)**

10.1.3 Parameter Menus

The left-hand pane of BCP's main window contains menus for various instrument parameters. Most of these parameters will never have to be changed by the user, but some values (e.g., the detector beam center) may need to be updated from time to time. If changes are made, the parameters are updated by clicking the **Save** button .

The "Instrument" Category

This menu contains the configuration parameters for the goniometer and generator. These are set prior to installation and should not be changed by the user.

The "Generator" Category

If the software has control over the generator, this menu contains the power settings and limits for the generator. Otherwise, most of these parameters are accessible from the generator's I/O panel.

- The "Tube Limits" area contains the generator's power limit parameters. These values are set up prior to or during installation and should not need to be changed.
- The "Preferred" area contains the values for the standard user settings. If "user settings" are selected when setting the generator power in BCP, the generator will ramp up to the preferred values. The warm-up delay is the time delay between when the generator ramps up to the user settings and when the first image can be taken. The default should be 120 seconds (00:02:00 hr:min:sec).
- The "Standby" area contains target parameters for the generator if it is idle for the prescribed time. The default power settings are 45 kV and 5 mA. The bias can remain at the optimum setting. If desired, the idle time counter can be disabled during weekday hours.

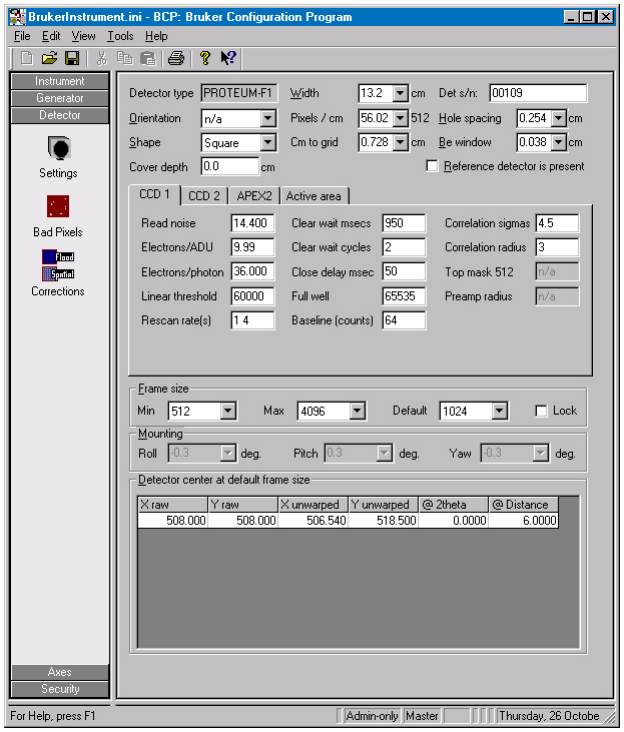
  Some users may want to disable the "Standby" feature. To do this, set the idle time to 3 days (72 hours) or longer, and the activate the **Exclude weekday** checkbox.

10.1.4 Change the Detector's Beam Center

1. Under the "Detector" category in BCP's left-hand pane, click **Settings**.

   The detector settings information appears.

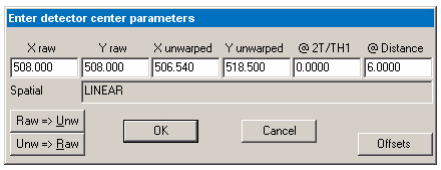
Figure 10.2 —Detector settings



2. In the “Detector center at default frame size” area, right-click on any of the values and choose **Modify**.

   The “Enter detector center parameters” window opens.

Figure 10.3 —“Enter detector center parameters” window



3. Enter the desired values for Xraw and Yraw. Click **OK**.
4. If you have a detector that requires a spatial correction, click the **Raw => Unw** button.

   If this button is disabled (i.e., grayed-out), load the spatial correction table under **Tools > Spatial** and try again.
5. Save the changes by choosing **File > Save**.

10.1.5 Collect a Dark Current Correction

CCD detectors require appropriate dark current frames before collecting data, and before using other BCP detector functions (e.g., Bad Pixels, Flood, and Spatial). Remember to collect dark current frames for all frame sizes.

NOTE: Be sure that the detector is cooled to its normal operating temperature before collecting a dark current correction.

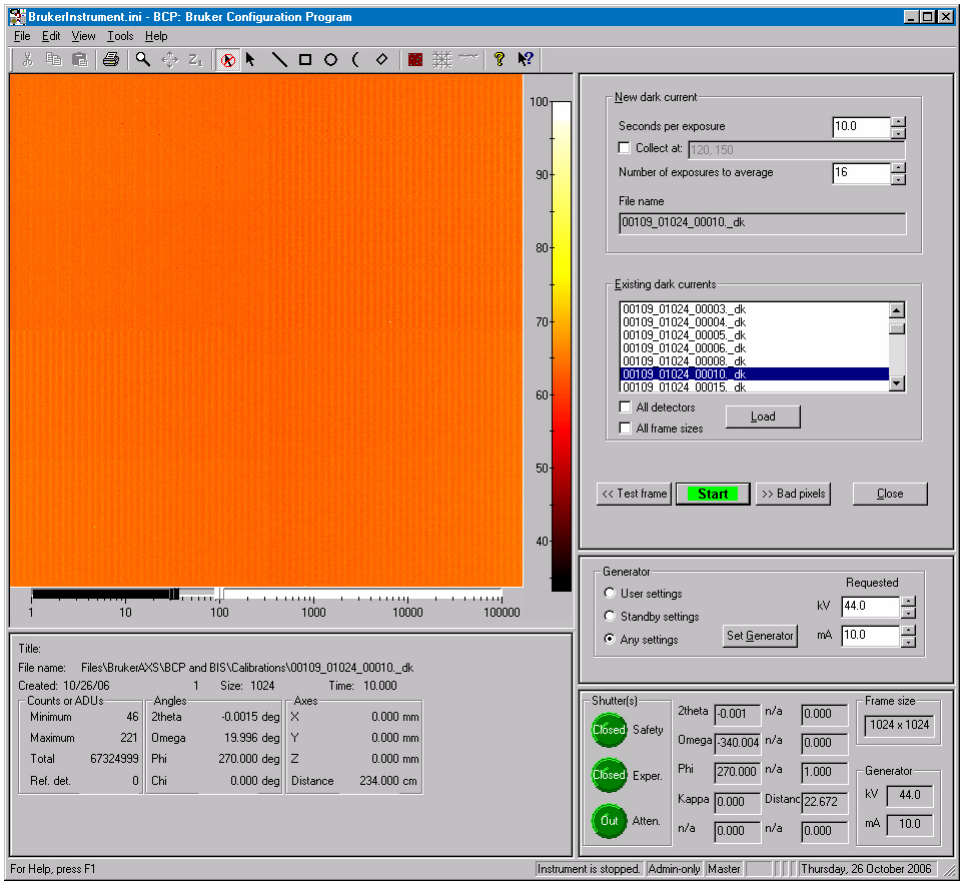
1. Choose **Tools > Dark Current**.
2. Set the “Seconds per exposure” value to the same time needed by your experiment (or your bad pixel, flood, or spatial frame).
3. Set the “Number of exposures to average” value to 9 (9 to 16 is recommended).

   This setting helps to reduce noise.
4. Click **Start**.

   The filenames are determined by BCP, and the files are stored in the “/Calibrations” folder.

   As each image starts, a message displays on the status line. When all images have been collected and averaged, the final averaged image appears.

Figure 10.4 —Dark current collected



10.2 Set Default Orientation Matrix Scans in the bn-config.py File

The bn-config.py file contains default orientation matrix run settings.

1. Open the bn-config.py file in a text editor.
2. Edit the following parameters (or, if they are not present, add them at the end of the file):
    2.1. Set the 2theta angle using the parameter `perpendicular_matrix_runs_2theta`.
    2.2. Set the detector distance using the parameter `perpendicular_matrix_runs_distance`.
    2.3. Set the extent of the scan using the parameter `perpendicular_matrix_runs_sweep`.
    For example, a value of `3` for this variable would mean that six 0.5° images would be collected for a total of 3°.
    2.4. Set the number of scans to collect using the parameter `perpendicular_matrix_runs_nr_sweeps`.
    If more than one scan is requested, the program uses different settings of the phi axis for each scan.
    2.5. Set the default exposure time using the parameter `perpendicular_matrix_runs_frametime`.
3. Save the bn-config.py file, and exit the text editor.

NOTE: If APEX2 is running when the bn-config.py file is updated, it should be restarted so that it reads the updated bn-config.py file.

10.3 Face Indexing with the Crystal Faces Module

In order to perform numerical correction for intensity data, the crystal's faces must be indexed. This allows a very exact crystallographic description of the crystal's shape.

Once you have a unit cell for your crystal, the Face Indexing module allows you to define faces directly over images of the crystal. As you define more and more faces, The module automatically determines the dimensions of the crystal.

This example uses a large crystal for which the unit cell has already been determined.

10.3.1 Starting the Crystal Faces Module

1. From the Task Bar's "Scale" category, click the **Crystal Faces** icon .

   The Crystal Faces module starts.

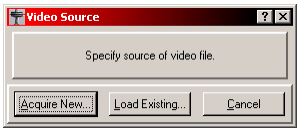
10.3.2 Collecting a Video Zip Stream

To index the faces, first collect a *video zip stream*, which is a series of images taken with the video microscope as the crystal is rotated through 360° about the phi axis.

NOTE: For the best results, make sure that lighting conditions are optimal for the crystal you are viewing. If possible, try to illuminate the crystal for optimum contrast between the crystal and the background. Also, it may desirable to use multiple light sources to illuminate several faces simultaneously.

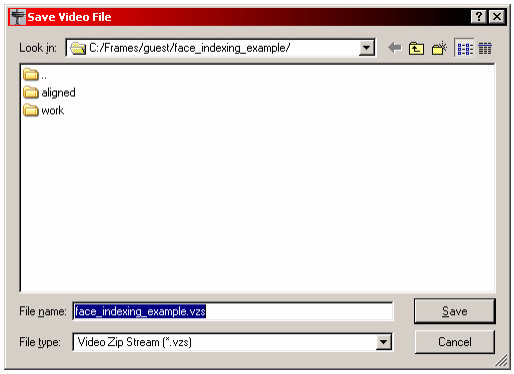
1. If no video zip stream has been collected for this crystal, a dialog appears prompting you to acquire a new video zip stream or load an existing one.

Figure 10.5 —"Video Source" window



2. Because we have not yet taken a video zip stream for this crystal, click **Acquire New...**. The “Save Video File” dialog (defaulted to your current project directory) appears.

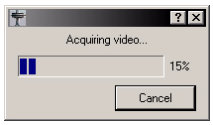
Figure 10.6 —“Save Video File” dialog



3. Click **Save**.

   If VIDEO is not open, it will open with a progress bar showing the video zip stream collection. When the video zip stream is fully collected, the Face Indexing initial view will appear.

Figure 10.7 —Progress bar



10.3.3 The Crystal Faces Module Interface

The Face Indexing module uses a dial at the bottom of the screen to step through the images of the crystal, and a tool called the T-tool to define faces. The Face List, shown on the right, displays the faces you have defined for the crystal and their distance from the instrument center.

Figure 10.8 —Crystal Faces module: initial view

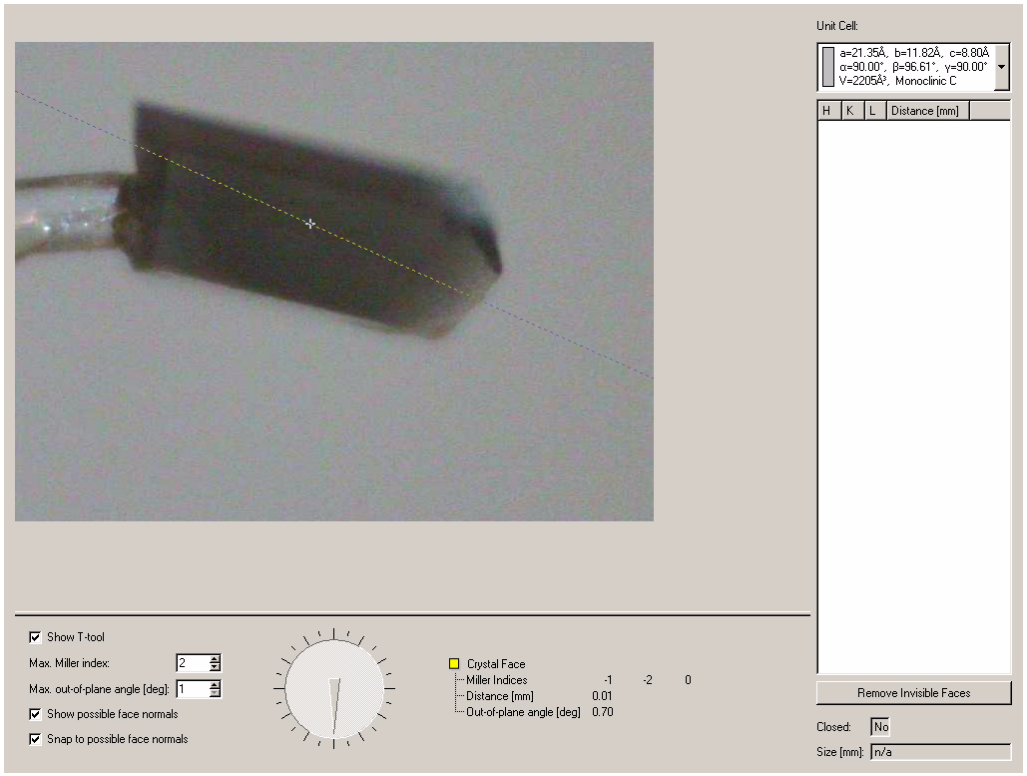


Table 10.1 — Face indexing controls

Item	Description
Show T-tool	Show and hide the T-tool for a better view of the crystal faces.
Max. Miller index:	The highest Miller index shown as face normals in the overlay.
Max. out-of-plane angle (deg):	When the T-tool snaps to a face normal, the plane you define may differ from the specified index by a maximum of this amount.
Show possible face normals	The module suggests face normals.
Snap to possible face normals	The T-tool snaps to the suggested face normals.
Dial (or mouse wheel)	Use the dial to rotate around 360° of phi. The right-click menu lets you rotate phi by 180° to define parallel faces.
Crystal face information	Shows the Miller indices of a given face, the face's distance from the instrument center, and the out-of-plane angle.

Table 10.2 — Face list controls

Item	Description
Unit Cell	Displays the unit cell that the module uses to display faces in the overlay.
H,K,L	Miller indices of the normal to the specified face.
Distance (mm)	The specified face's distance to the center of the instrument.
Remove Invisible Faces	Faces obscured by the body of the crystal are removed from the Face list.
Closed:	Displays whether the faces you have defined encapsulate a closed volume.
Size (mm):	Dimensions of the crystal, calculated according to the defined faces.

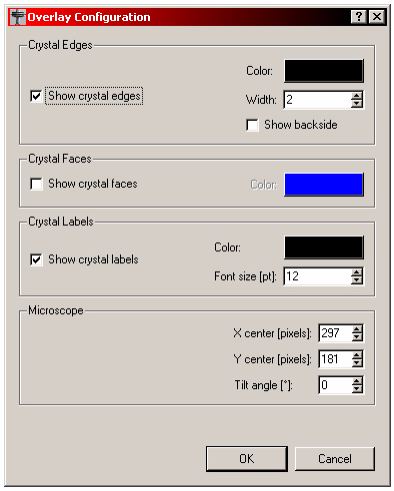
Setting View Options

Depending on the color and reflectivity of the crystal and background, you may want to adjust the display colors.

1. Right-click in the image display area, and choose **Configure Overlay** from the menu.

   The "Overlay Configuration" window appears (Figure 10.9).

Figure 10.9 —"Overlay Configuration" window

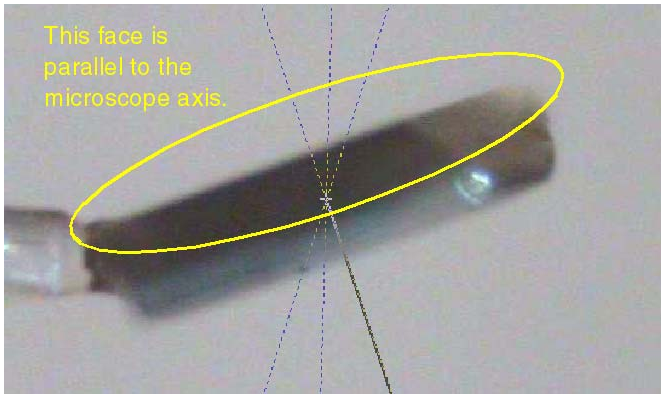


10.3.4 Specifying Crystal Faces

1. Use the dial control at the bottom of the screen (or use the mouse wheel) to rotate the crystal until one of the faces is parallel to the microscope axis as in Figure 10.10.

   Note that, because **Show T-Tool** and **Show possible face normals** are activated, the module suggests possible faces (whose indices are within the Max. Miller Index parameter) with dotted lines.

Figure 10.10 —Face parallel to microscope axis

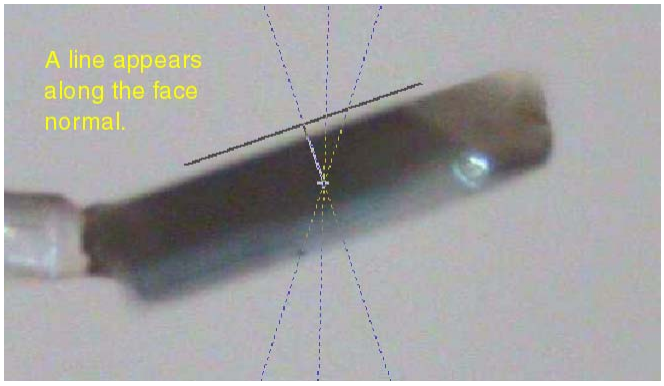


2. Move the mouse cursor to the dotted line that is perpendicular to the face.

   The mouse cursor snaps to the possible face normal. A line appears to show the possible face. This line defines a plane along the microscope axis, which should be parallel to the crystal face.

3. Use the mouse to move the line until it touches the crystal face.

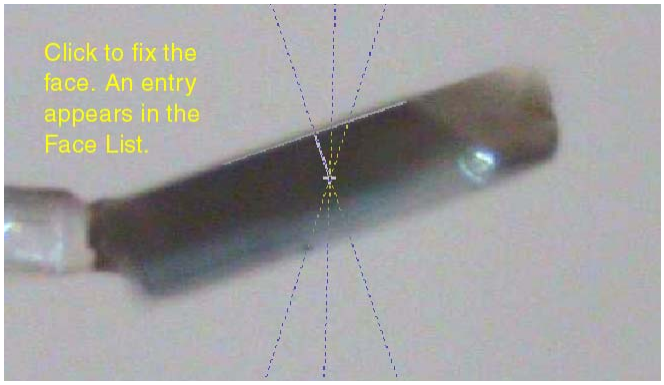
Figure 10.11 —Face normals



4. Click to fix the face.

   An entry appears in the Face List showing the Miller Indices of the crystal face, along with the face's distance from the instrument center.

Figure 10.12 —Fixing the face



5. Repeat the steps in this Section to specify all of the crystal's visible faces.

   If the module does not suggest a face normal for a certain face, you can increase the value in the "Max. Miller index" field to show more suggested normals. As you define more faces, the module will begin to display its geometric model of the crystal, superimposed over the video images (Figure 10.13).

Figure 10.13 —Some—but not all—faces defined

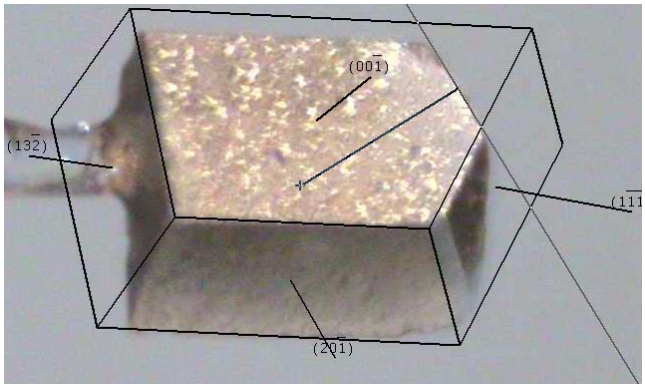
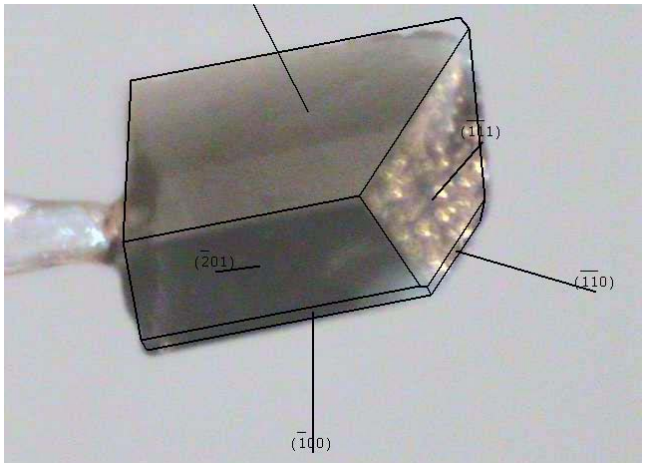


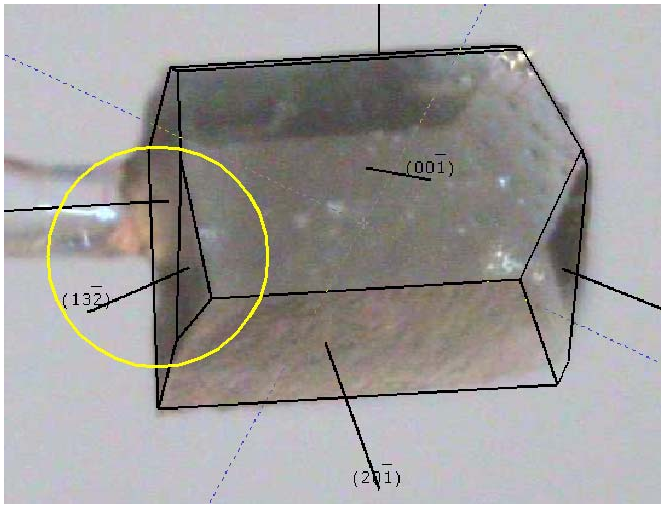
Figure 10.14 —All faces defined



10.3.5 Editing the Face List

Consider Figure 10.15. The face (1 3 -2) does not appear to define a face correctly. Removing this face will give a more accurate model of the crystal.

Figure 10.15 —Face (1 3 -2) is incorrect



1. In the Face List, select the HKL (1 3 -2).
2. Right-click and choose **Remove**. The face is removed from the list and the model is improved (Figure 10.17).

NOTE: Alternatively, you can select **Clear List** to start over, or **Add** and **Edit** to define your own faces and see where they appear on the crystal.

Figure 10.16 —Face List right-click menu

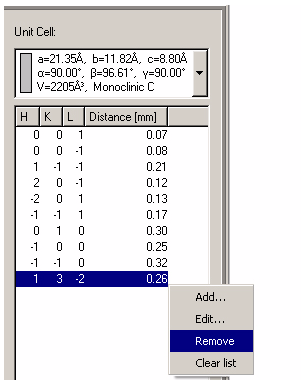
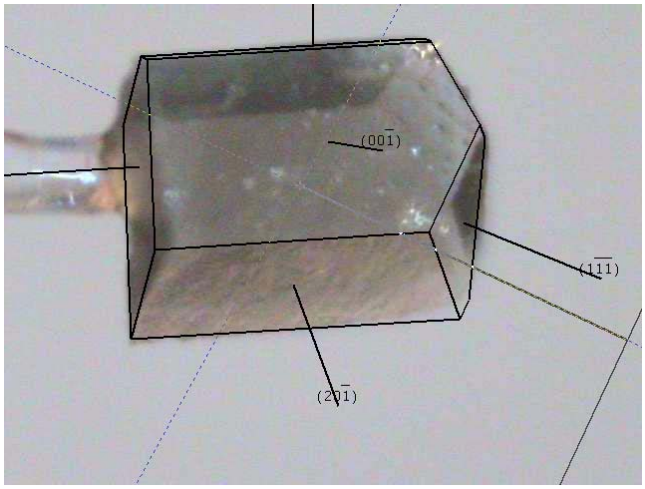


Figure 10.17 —Face (1 3 -2) removed



10.3.6 Scaling with Face Indices

1. When all faces are defined, the module has accurate crystal dimensions (as shown in the bottom right-hand corner). At this point, you can exit the Face Indexing module and proceed with scaling.
2. In the Scale module, be sure to select **Numerical Absorption Correction** under the “Setup” tab.

10.4 The Reciprocal Lattice Viewer Module

The Reciprocal Lattice Viewer module is a very powerful tool for viewing the harvested reflections in a reciprocal lattice.

10.4.1 Start the Reciprocal Lattice Viewer Module

1. In the Task Bar's "Evaluate" section, click the **Reciprocal Lattice Viewer** icon .

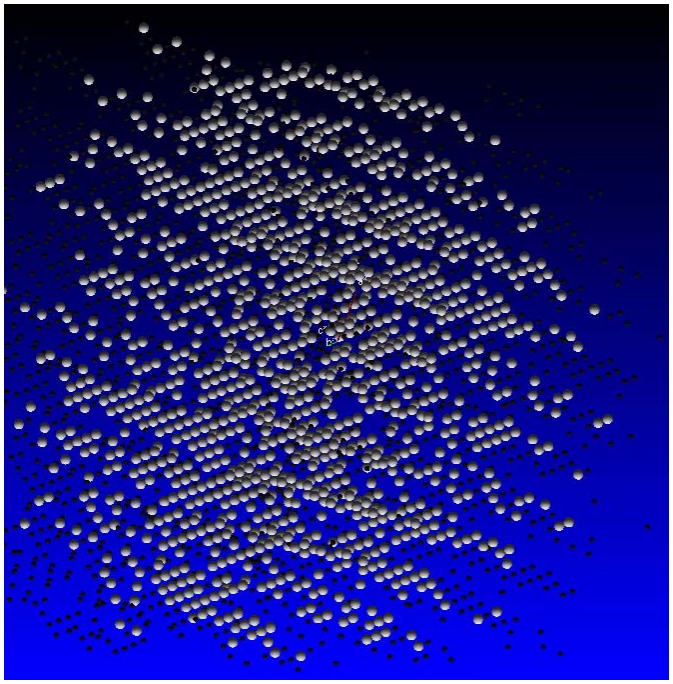
   The Reciprocal Lattice Viewer module opens.

   The module automatically reads the reflections from the current project and displays them as a reciprocal space plot.

10.4.2 Views and Tools

Figure 10.18 is a typical unoriented view after import. The gray circles are more intense reflections, i.e., those with higher I/sigma(I). The black dots represent less intense reflections. The intensity key slider at the bottom of the window can be used to change the selection criterion.

Figure 10.18 —Unoriented view



Other tools can be accessed by right-clicking on the background to give the “Quick Tools” menu (Figure 10.19), and by choosing **RLATT** in the Menu Bar (Figure 10.20).

Figure 10.19 —Quick Tools menu

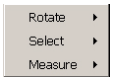
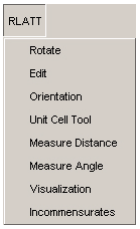


Figure 10.20 —Full menu tools

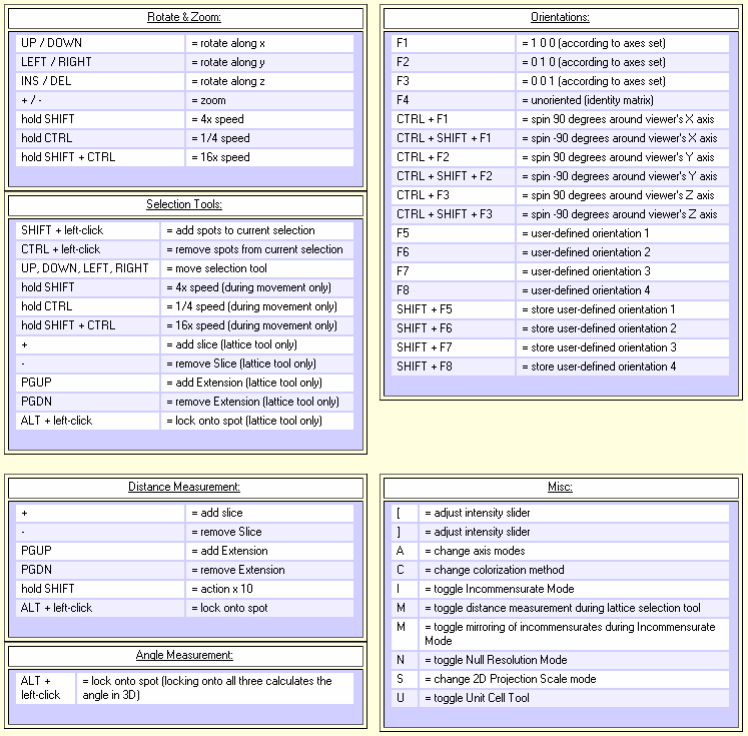


Additional context-sensitive information is available by clicking the “What’s This” help arrow in the Menu Bar.

Figure 10.21 —“What’s This?” help icon

For example, clicking the “What’s This?” help icon and then clicking in the work area (i.e., the gray area to the right of the lattice display) gives the shortcuts shown in Figure 10.22. Clicking on the blue background gives the hints shown in Figure 10.23.

Figure 10.22 —Shortcut keys and commands



Rotate & Zoom:

UP / DOWN	= rotate along x
LEFT / RIGHT	= rotate along y
INS / DEL	= rotate along z
+ / -	= zoom
hold SHIFT	= 4x speed
hold CTRL	= 1/4 speed
hold SHIFT + CTRL	= 16x speed

Selection Tools:

SHIFT + left-click	= add spots to current selection
CTRL + left-click	= remove spots from current selection
UP, DOWN, LEFT, RIGHT	= move selection tool
hold SHIFT	= 4x speed (during movement only)
hold CTRL	= 1/4 speed (during movement only)
hold SHIFT + CTRL	= 16x speed (during movement only)
+	= add slice (lattice tool only)
-	= remove Slice (lattice tool only)
PGUP	= add Extension (lattice tool only)
PGDN	= remove Extension (lattice tool only)
ALT + left-click	= lock onto spot (lattice tool only)

Orientations:

F1	= 1 0 0 (according to axes set)
F2	= 0 1 0 (according to axes set)
F3	= 0 0 1 (according to axes set)
F4	= unoriented (identity matrix)
CTRL + F1	= spin 90 degrees around viewer's X axis
CTRL + SHIFT + F1	= spin -90 degrees around viewer's X axis
CTRL + F2	= spin 90 degrees around viewer's Y axis
CTRL + SHIFT + F2	= spin -90 degrees around viewer's Y axis
CTRL + F3	= spin 90 degrees around viewer's Z axis
CTRL + SHIFT + F3	= spin -90 degrees around viewer's Z axis
F5	= user-defined orientation 1
F6	= user-defined orientation 2
F7	= user-defined orientation 3
F8	= user-defined orientation 4
SHIFT + F5	= store user-defined orientation 1
SHIFT + F6	= store user-defined orientation 2
SHIFT + F7	= store user-defined orientation 3
SHIFT + F8	= store user-defined orientation 4

Distance Measurement:

+	= add slice
-	= remove Slice
PGUP	= add Extension
PGDN	= remove Extension
hold SHIFT	= action x 10
ALT + left-click	= lock onto spot

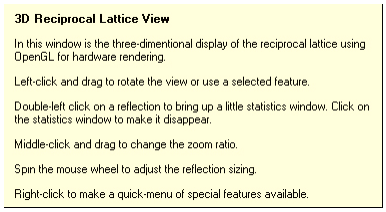
Angle Measurement:

ALT + left-click	= lock onto spot (locking onto all three calculates the angle in 3D)

Misc:

[	= adjust intensity slider
]	= adjust intensity slider
A	= change axis modes
C	= change colorization method
I	= toggle Incommensurate Mode
M	= toggle distance measurement during lattice selection tool
M	= toggle mirroring of incommensurates during Incommensurate Mode
N	= toggle Null Resolution Mode
S	= change 2D Projection Scale mode
U	= toggle Unit Cell Tool

Figure 10.23 —3D display information window



3D Reciprocal Lattice View

In this window is the three-dimentional display of the reciprocal lattice using OpenGL for hardware rendering.

Left-click and drag to rotate the view or use a selected feature.

Double-left click on a reflection to bring up a little statistics window. Click on the statistics window to make it disappear.

Middle-click and drag to change the zoom ratio.

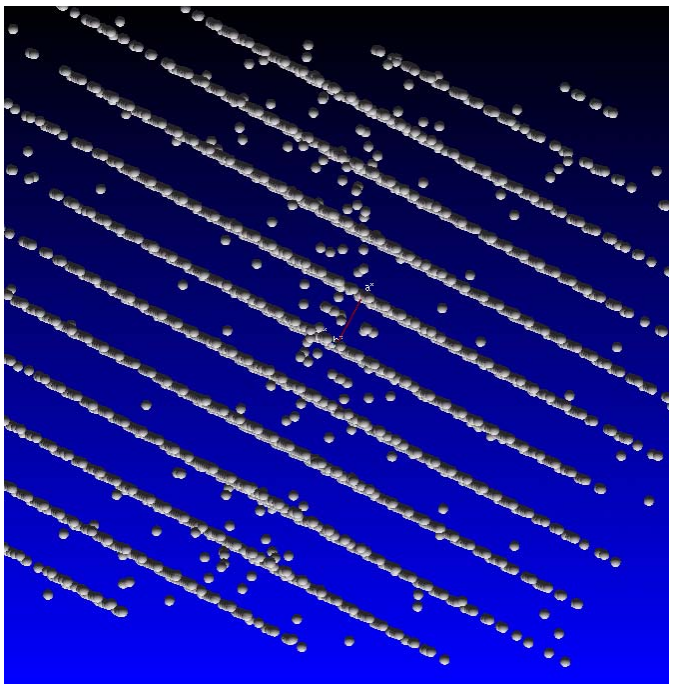
Spin the mouse wheel to adjust the reflection sizing.

Right-click to make a quick-menu of special features available.

Orienting Views

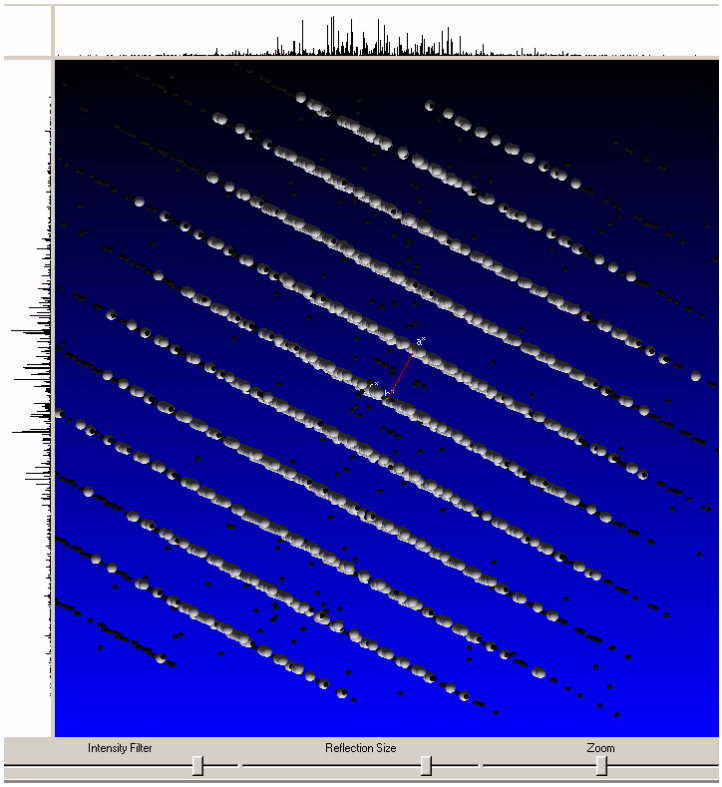
Move the mouse to rotate the lattice display. It is possible to easily see rows and non-fitting peaks (see Figure 10.24).

Figure 10.24 —Display looking down layers of reflections



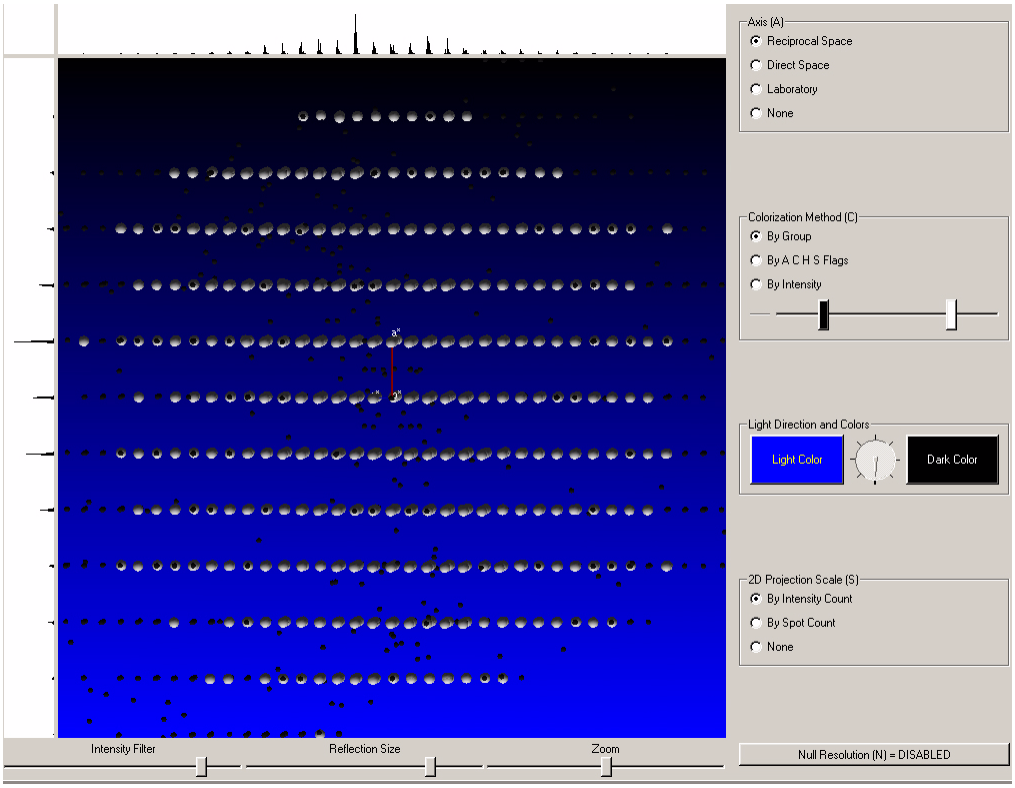
By moving the intensity slider at the bottom of the display, it is easy to deselect weak reflections. In Figure 10.25, the reflections between the layer lines all turn black when the intensity filter is moved to the left.

Figure 10.25 —Display looking down layers of reflections. Weaker reflections, i.e., those with smaller I/sigma(I), are black because the “Intensity Filter” slider has been moved to the left.



With additional rotations, the layers of reflections can be further oriented so that stacks of reflections become visible. Sometimes it is quite useful to right-click on the background and choose Z-rotations from the rotation options. The 2D profiles on the top and to the left are a valuable aid in this process. With practice, the 2D profiles can be organized into clusters. As shown, the 2D profiles are counting intensity. The counting mode can be changed to spot count or turned off completely using the Visualization menu (**RLATT > Visualization**) or by pressing the [s] key to cycle through the counting modes.

Figure 10.26 —An oriented lattice view. The Visualization menu is to the right. The 2D profiles are to the left and to the top.



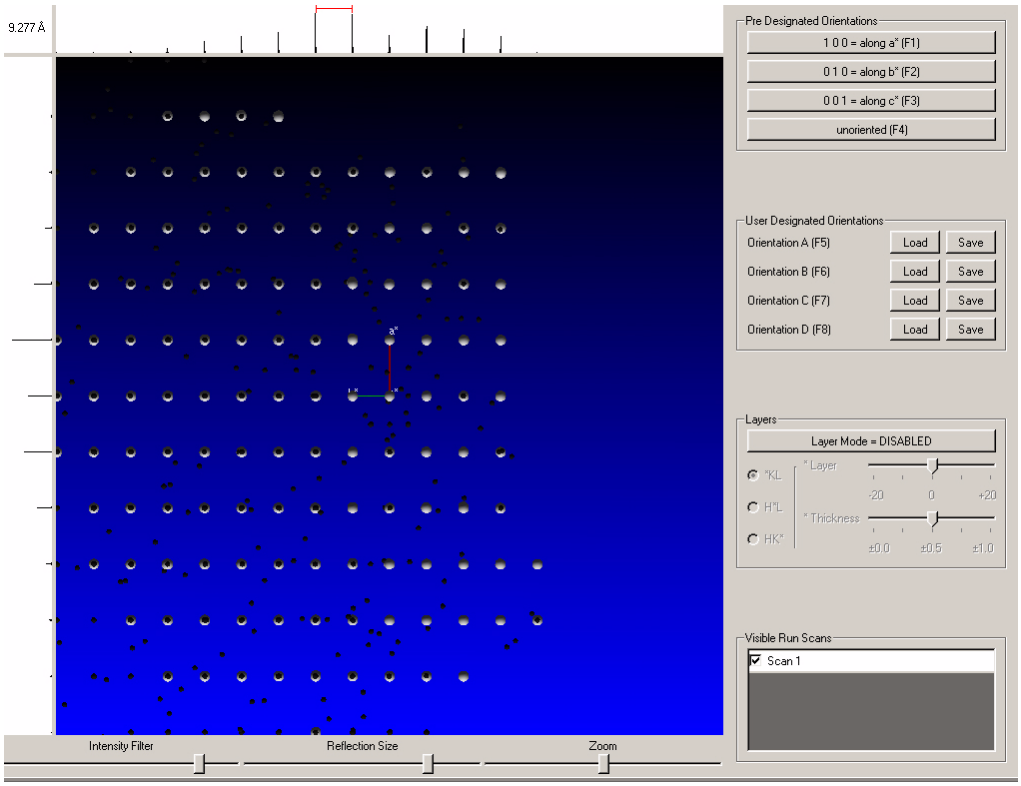
The distance between layers can be measured by clicking on one of the clusters of lines in the 2D view and then dragging to the next cluster. If you drag over three clusters, then the distance as measured would need to be multiplied by three.

Figure 10.27 —Horizontal measured distance

Figure 10.28 —Vertical measured distance

The Orientation menu allows easy orientation if the cell is indexed. Press the [F1], [F2], or [F3] key or click the appropriate button.

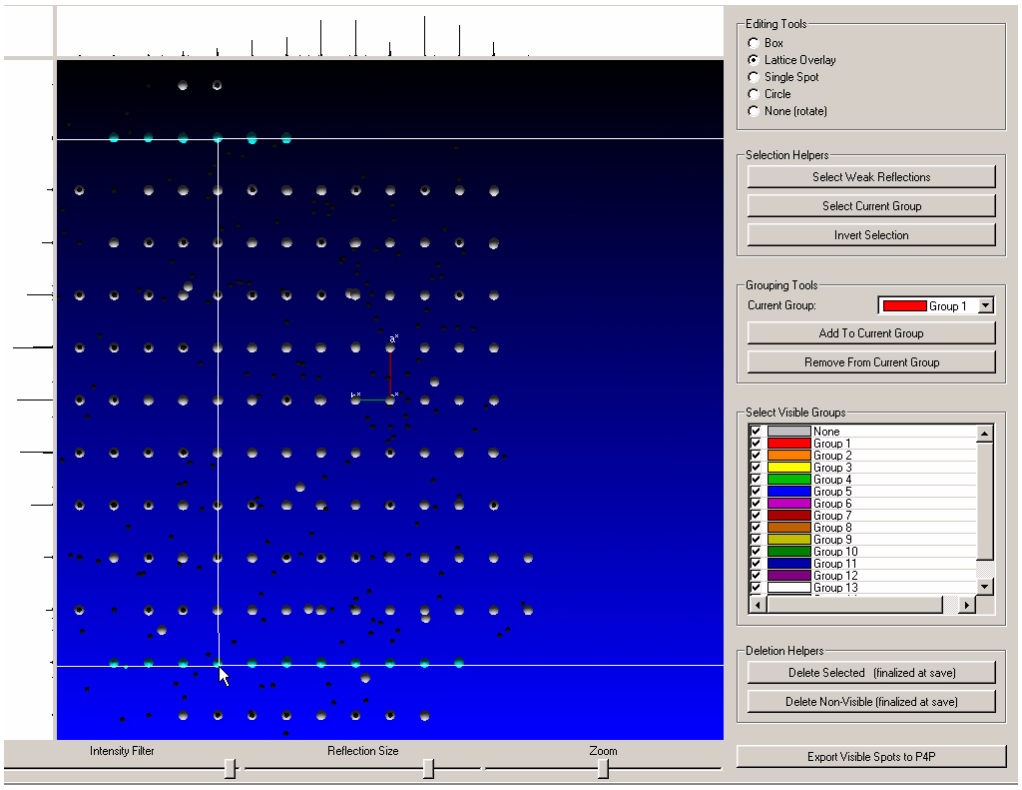
Figure 10.29 —Orientation menu with aligned view



The Lattice Tool

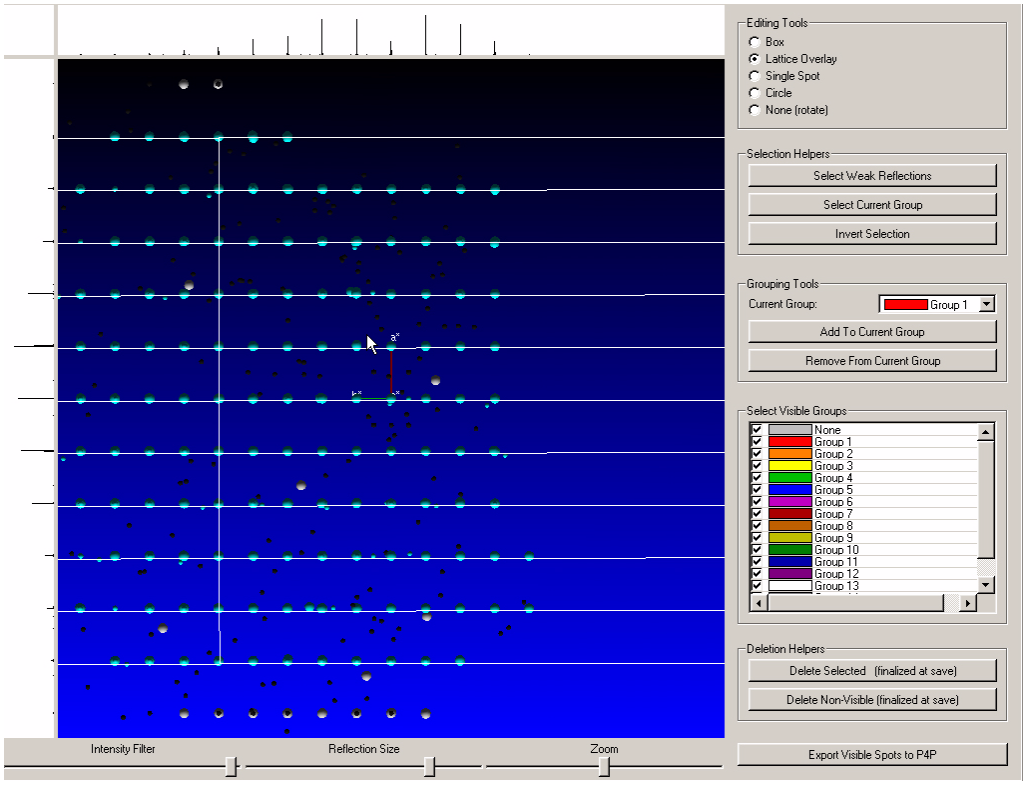
The Lattice tool is one of the most useful editing tools. Activate it from the Edit menu or by clicking on the background and choosing **Select > Lattice**. When initially activated, nothing appears. Hold down the [Alt] key to lock on a centroid and then left-click and hold on a spot. Now drag the mouse and line up the line that is tied to your spot on a row. Choose a longer row and align carefully. Now, while still holding the left mouse button, drag the second line to another row as shown in Figure A.13. Let go of the button. If you want to start over, click on another spot and repeat the process. Using the [Alt] key is optional, but it makes alignment of the lines easier and more accurate (Linux users may find that the [Alt] key has operating system functions).

Figure 10.30 —Two lattice lines selected



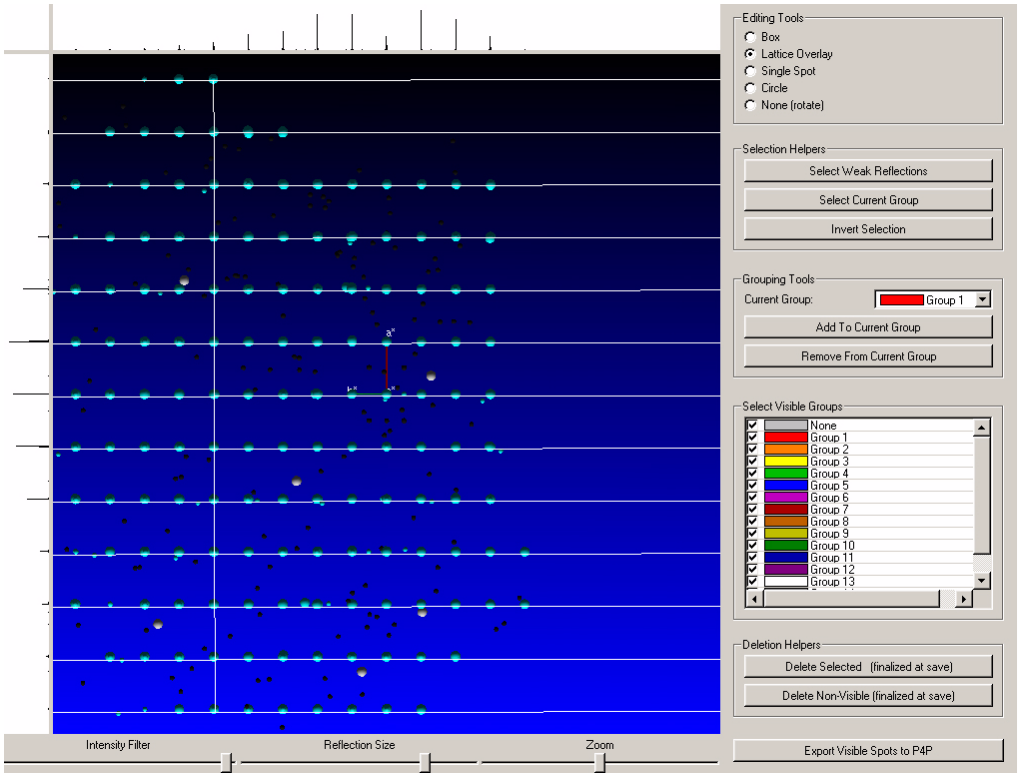
Press the [+] key to add lines between the two you have marked. Press the [-] key to remove lines.

Figure 10.31 —View with most of the lattice lines selected (lines to the top and bottom are not selected).



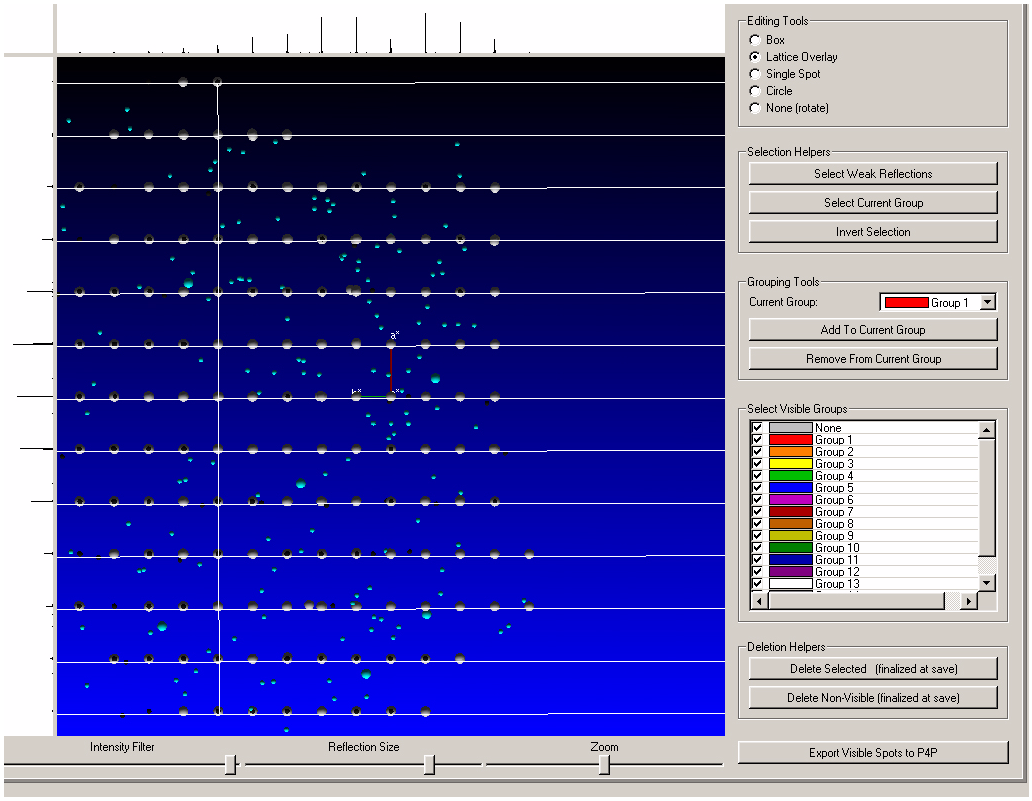
Use the [Page Up] key to add lines to the outside of the previously selected lines (in this example, the top and bottom). [Page Down] removes lines from the outside. Now all visible lines are selected. Note that the selected reflections are turquoise.

Figure 10.32 —A view with all the layers selected



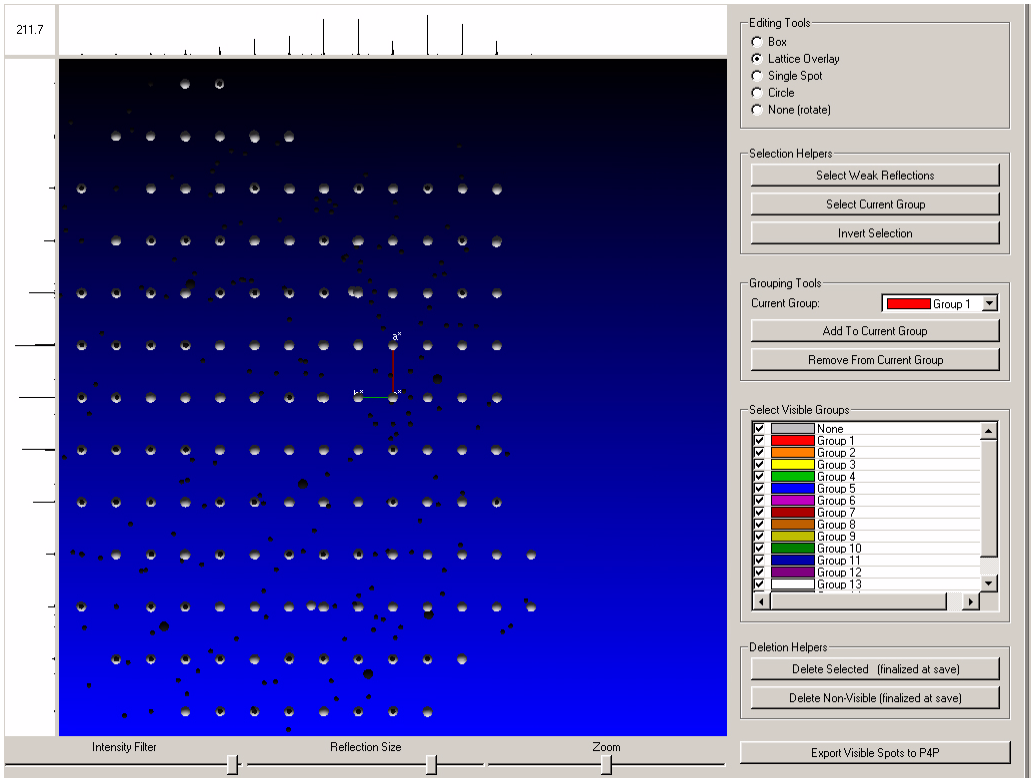
Click **Invert Selection** (in the “Editing Menu” (**RLATT > Edit**) under “Selection Helpers”). All of the spots that do not touch the layer lines are now selected.

Figure 10.33 —Spots selected between layers



Click **Delete Selected (finalized at save)** to mark the selected reflections to be deleted (and turned black). Alternatively, these selected reflections can be added to a group and then hidden using **Select Visible Groups**.

Figure 10.34 —Selected reflections are black



Save the file by clicking the disk icon , or use **File > Save** to remove the reflection from the reflection list. Hiding a group with **Select Visible Groups** does not remove it from the reflection list. Either way, the resulting image is much cleaner and easier to interpret.

The Unit Cell Tool

Enable the Unit Cell tool to put a colored box in the view. This allows you to see if reflections are actually falling on the corners of the box (see Figure 10.35 and Figure 10.36). There are multiple options in the Unit Cell tool. **Select a Visible Plane** displays individual planes. **Select Plane Size** determines the boundaries of the planes and the number of unit cells displayed. **Grid Planes** mode displays planes as grids with spacings determined by the lattice.

Figure 10.35 —Unit Cell tool enabled

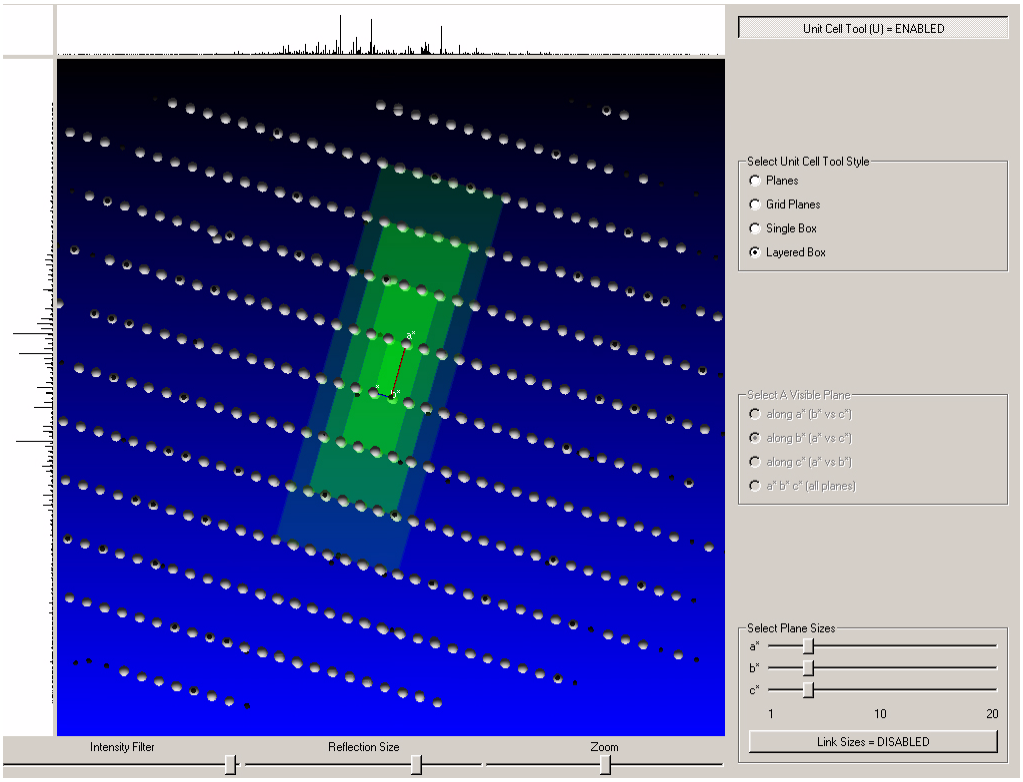
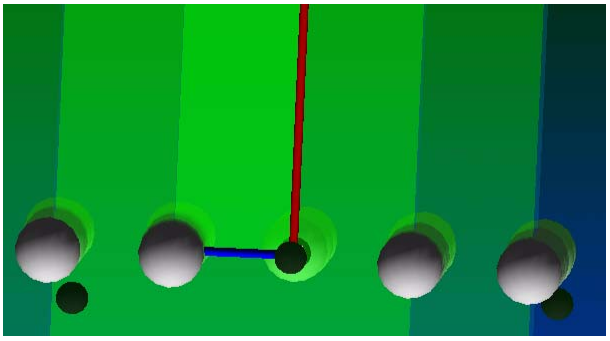


Figure 10.36 —A view that zooms in on the Unit Cell tool. Almost all spots lie on the lines and planes defined by the unit cell. To the left and right are two weaker reflections (black dots) that do not fit.



10.4.3 Defining Groups

Selected reflections can be grouped. This tool is most useful for examining twinned, split, ingrown, and otherwise problematic crystals. For Figure 10.37, alternate rows were selected with the Lattice Selection tool. In the Grouping Tools section of the tool boxes to the right, the current group was set to Group 1 (red). Click “Add to Current Group” to turn these rows red. Then, the other rows and Group 13 (white) were selected and added. The box tool was used to select the reflection to the top left and these were put in Group 3, the yellow group. Finally, some of the reflections to the top and bottom were selected and added to Group 5, the blue group. This example of using the color groups generates a flag-type display. For twinned crystals, etc., the groups would be used to denote different components (Figure 10.38).

Figure 10.37 —Reflections selected for different groups

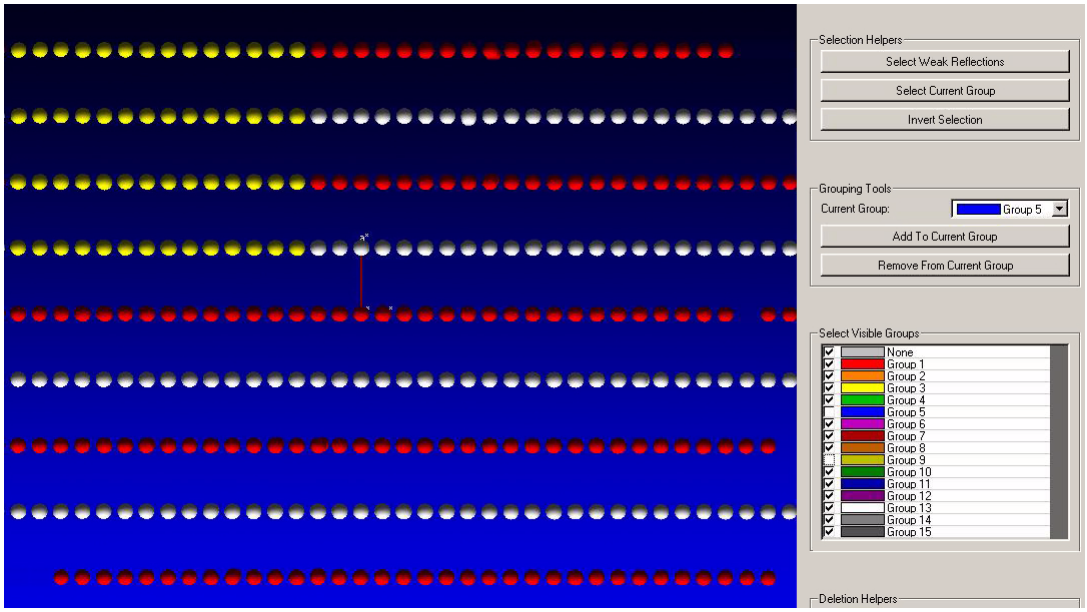
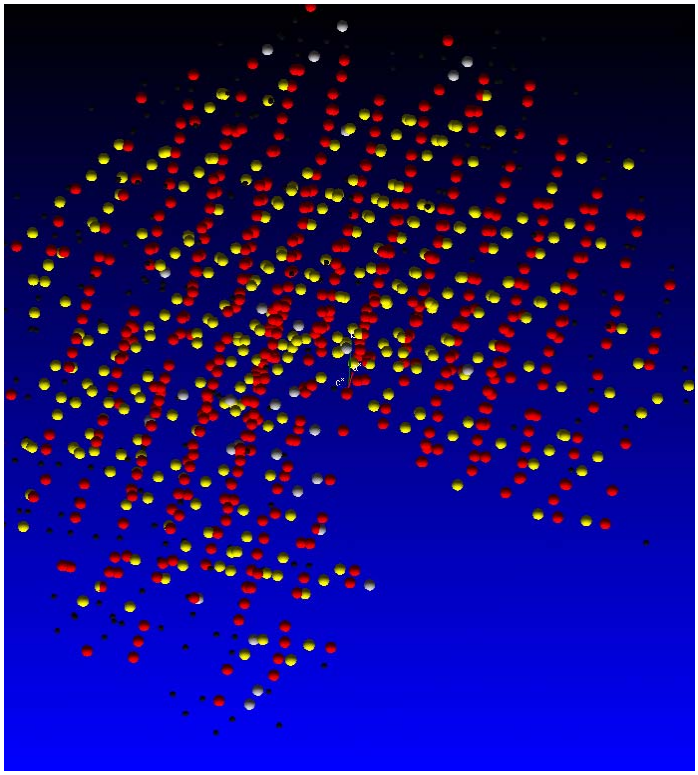


Figure 10.38 — A more practical use of the selection and color groupings. The two components of a rotational twin are colored yellow and red.

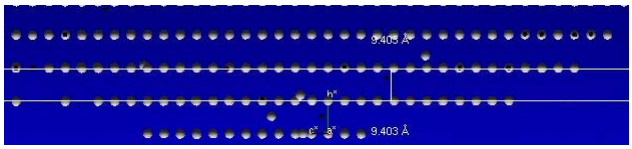


10.4.4 Measuring Distances and Angles

Right-click on the background and choose **Measure** to get tools for measuring distances and angles.

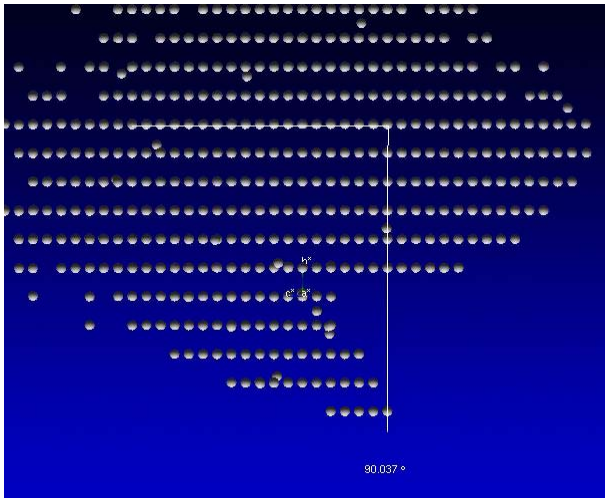
The Measure Distance tool gives two lines, much like the lines in the Lattice Selection tool, that can be oriented and dragged to get lattice spacings (Figure 10.39). Use the [Alt] key to lock on spot centers for easier orientation and more accurate measuring.

Figure 10.39 —Measure distance tool



With the Measure Angle tool, left-click, hold, and drag a line on a lattice layer ending on the spot that will become the vertex of the angle. When the mouse is released, that point becomes the pivot point for a new line. Moving the mouse with no buttons depressed gives angle measurements (Figure 10.40). As described, the angle measurements will be done in 2D mode (i.e., the angle between the two lines displayed). To measure an angle in 3D mode, hold the [Alt] key while selecting spots as above. The tool will then lock on to spot centroids and the angle will be calculated using spot coordinates. This eliminates errors that might result from measuring using the 2D projection of the current orientation.

Figure 10.40 —Measure angle tool



10.4.5 Writing a .p4p File

At the bottom of the **Edit** menu, there is a button for exporting all visible reflections to a .p4p file. The .p4p file is a text file of crystal, instrument, and reflection information. If the cell and crystal orientation is known, that information is included also (otherwise, dummy values are included).

The sequence of cleaning up a set of reflections using the Reciprocal Lattice Viewer module and then exporting the results for input to CELL_NOW is a useful tool for dealing with hard-to-index crystals.

10.5 Using CELL_NOW

CELL_NOW is an extremely powerful tool for determining unit cells for difficult crystals. It analyzes a list of reflections to find a cell and orientation matrix despite the presence of several twin domains or other problems.

In initial search mode, the program tries to find sets of reciprocal lattice planes that pass close to as many reflections as possible. The corresponding real space vectors are sorted on a figure of merit (1.0 being a perfect fit). After the vector list has been output, CELL_NOW attempts to suggest a suitable cell. This will not necessarily be the conventional cell, so it should be checked using XPREP.

Once a cell is found, it may be rotated to locate further twin domains iteratively using only the reflections that have not yet been indexed.

If CELL_NOW fails to suggest a sensible cell, either something is seriously wrong with the reflection list (e.g., a wrong detector distance) or a cell axis is longer than the given search range.

10.5.1 Running CELL_NOW on a List of Reflections

1. Open a Command Prompt window.
2. Navigate to the directory containing the exported reflection list.
3. For Windows or Linux, enter the command `cell_now`.

   The terminal prints out an explanation of the program. Read this as it has useful information about what the program is doing:

   ```
   CELL_NOW analyses a list of reflections to find a cell and
   orientation matrix despite the presence of several twin domains
   or other junk. In initial search mode the program tries to find
   sets of reciprocal lattice planes that pass close to as many
   reflections as possible. The corresponding real space vectors are
   sorted on a figure of merit (1.0 would be a perfect fit). In the
   output these are followed by the percentages of reflections that
   fit within 0.1, 0.2 and 0.3 times the interplanar separation, the
   components a1, a2 and a3 of the vector, the angles to previous
   vectors and a cross figure of merit to previous vectors. The
   latter should be larger for reflections belonging to the same
   twin component. Cosines of angles between vectors a and b can also
   be calculated from the components by (a1*b1+a2*b2+a3*b3)/(a*b).

   After the vector list has been output, CELL_NOW attempts to
   suggest a suitable cell. This will not necessarily be the
   conventional cell, so it should be checked using XPREP (without
   an .hkl file) taking the lattice type found with CELL_NOW into
   account. If necessary this conventional cell may be reinput in
   'specified cell' search mode to find the orientation matrix. If
   CELL_NOW fails to suggest a sensible cell, either something is
   seriously wrong with the reflection list (e.g. a wrong detector
   distance) or a cell axis is longer than the given search range.
   ```

```
In specified cell search mode the program tries to find the best
cell within the specified ranges. The reflections that fit this
cell within a specified fraction of all ?hree interplanar
spacings may be flagged as indexed, and a new .p4p or .spin file
written in which they have the 'H' flag so that they can be
displayed in a different color with RLATT. Then the cell may be
rotated to locate further twin domains iteratively using only the
reflections that have not yet been indexed.

 ** WARNING: the exhaustive search employed in this program is
VERY SLOW **
** so a CPU clock frequency of AT LEAST 3GHz is strongly
recommended **
```

4. CELL_NOW requests the name of the input file and suggests a name for the output file. Enter the name of the input file (For the output file, press [Enter] to accept the default).

```
Full name of .p4p, .spin or .drx file to read: example.p4p

999 reflections read in
Listing file [example._cn]:

514 reflections read in
Listing file [example._cn]:
```

5. Press [Enter] to start a general search.

```
Initial search (<Enter>) or specified cell search (S):
```

6. If the default superlattice threshold is suitable, press [Enter].

```
Superlattice threshold: an axis will be rejected if less than
this percentage of reflections has indices not equal to 2n or 3n
resp. [10]:
```

7. If the default minimum and maximum distances are acceptable, press [Enter] again.

```
Minimum and maximum allowed values for cell edge [5 40]:
```

NOTE: Usually CELL_NOW is used to search generally for a cell, but if the cell is known, choose the specified cell option and enter the cell dimensions. CELL_NOW will then search for a matching cell.

After some period of time, a list of real space vectors sorted on a figure of merit (1.0 being a perfect fit) will be output. These are followed by the percentages of reflections that fit within 0.1, 0.2 and 0.3 times the interplanar separation, the components a1, a2 and a3 of the vector, the angles to previous vectors and a cross figure of merit to previous vectors. The latter should be larger for reflections belonging to the same twin component. Cosines of angles between vectors a and b can also be calculated from the components by (a1*b1+a2*b2+a3*b3)/(a*b).

```
Length, fom, #hits, %(0.1), %(0.2), %(0.3), components
 Followed by angle/correlation to previous vectors:

   12.692  0.902    30    94.493  99.999  99.999   -0.475    5.493  -11.432

   11.016  0.891    20    99.098  99.698  99.899    0.491   -9.912   -4.782
  90.0
 0.798

   16.802  0.836    24    93.793  99.398  99.698   -0.011    4.417   16.211
 139.0 131.0
 0.754 0.755

   16.805  0.819    25    90.189  99.498  99.899    0.953  -15.402    6.654
 139.1  49.1  81.9
 0.749 0.739 0.688

   27.693  0.741    19    80.379  93.692  98.898   -0.446    1.111  -27.668
  23.4  66.6 162.4 115.7
 0.675 0.657 0.638 0.616

   27.701  0.736    43    78.477  92.091  97.296    1.396  -20.915   18.109
 156.6  66.6  64.4  17.5 133.2
 0.687 0.659 0.620 0.645 0.569

   25.428  0.722    45    77.376  98.497  99.598   -0.496   14.332   20.999
 119.9 150.1  19.1 101.0 143.3  83.5
 0.651 0.674 0.638 0.600 0.557 0.533

   18.381  0.688    35    72.271  86.285  91.791  -15.128    9.256    4.827
  89.3 127.1  67.3 113.8 103.2 104.5  58.9
 0.596 0.605 0.556 0.544 0.480 0.484 0.487

    6.329  0.686     4    65.465  65.465  65.465    0.248   -2.729    5.705
 179.9  90.1  40.9  41.0 156.7  23.5  60.0  90.7
 0.574 0.573 0.532 0.524 0.459 0.469 0.465 0.426

   14.648  0.684    18    73.873  85.484  91.090  -14.633   -0.652    0.025
  89.1  90.3  90.5  90.9  89.3  90.9  90.2  36.8  91.1
 0.591 0.603 0.556 0.539 0.475 0.477 0.490 0.539 0.424
.
.
.
-------------------------------------------------------------------------------
```

After the vector list has been output, CELL_NOW attempts to suggest a suitable cell. This will not necessarily be the conventional cell, so it should be checked using XPREP (without an .hkl file) taking the lattice type found with CELL_NOW into account. If necessary, this conventional cell may then be input in 'specified cell' search mode to find the orientation matrix.

```
The following cells would appear to be plausible, but should be checked using  XP
REP because they are not necessarily the conventional cells.

 FOM, % with i       n  0 .2, a..gamma, volume and lattice type for potential
unit-cells:
      1     1 .000    85.3   12.692  11.016  14.648   89.70   90.95   90.00
2047.7  P
-------------------------------------------------------------------------------
 Cell for domain  1:   12.692   11.016   14.648    89.70    90.95    90.00
 Figure of merit: 0.619   %(0.1):  68.3   %(0.2):  85.3   %(0.3):  91.0
```

CELL_NOW has identified a domain that fits 85.3% of the data with a tolerance of 0.2.

The output shows a suitable cell with its orientation matrix, figure of merit and the deviations of the input reflections from integer indices. This deviation is used to determine which reflections fit this cell and which are left for further searches for new domains.

```
Orientation matrix:  0.00164338 -0.00357404 -0.06816319
                    -0.03417112  0.08170117 -0.00385047
                     0.07098872  0.03940338  0.00098036

Maximum deviation from integer index [0.25]:
```

8. Enter the maximum deviation from integer index cutoff (Typically the default is reasonable). Press [Enter].

```
Percentages of reflections in this domain not consistent with lattice types:
A: 50.1,  B: 49.9,  C: 45.0,  I: 48.9,  F: 72.5,  O: 64.3  and  R: 65.8%
```

Lattice information (given as % of relevant reflections that do not fit a particular lattice) should be used in XPREP to determine the conventional cell. Typically, for a lattice to be present the % inconsistent should be less than 10, but problem crystals can make the value higher.

```
Percentages of reflections in this domain that do not have:
h=2n: 36.3,  k=2n: 49.8,  l=2n: 49.3,  h=3n: 69.1,  k=3n: 67.2,  l=3n: 66.4%
```

This percentage information is useful for identifying supercell problems. Typically, the percentages should be 30% or higher for the 2n values and 50% or higher for the 3n values, but problem crystals or crystals with heavy atoms in special positions can make the values lower. Low values for a particular class of reflections can suggest that an axis is double (2n) or triple (3n) its correct length.

```
New cell from list (number), reorientate (R), accept (A) or quit (Q) [A]: A
```

9. Enter A or just press [Enter] to accept this cell. The program asks for a name for a .p4p file. Enter the name of the .p4p file for this solution (in this example, domain1.p4p).

```
.p4p or .spin file to write domain to:domain1.p4p

RLATT color-coding employed in file: domain1.p4p
 White: indexed for first domain
 Red: not yet indexed

   877 reflections within 0.250 of an integer index assigned to domain  1,
   877 of them exclusively;   122 reflections not yet assigned to a domain
```

10. 122 reflections, approximately 1/10 of the data, have not yet been indexed (assigned to a domain). This is more than might be expected for random noise peaks. A search for an additional domain makes sense. The domain 1 cell will be rotated to locate further twin domains iteratively using only the reflections that have not yet been indexed. Enter `S` to search for another orientation of the newly chosen cell.

```
Re-refine initial cell (R), search for next domain (S), quit (Q) or choosenew cell
from list (enter number) [S]: S

Cell for domain  2:   12.692   11.016   14.648    89.70    90.95    90.00

 Figure of merit: 0.643   %(0.1):  69.7   %(0.2):  97.5   %(0.3):  98.4

 Orientation matrix:  0.00598966  0.00402327  0.06807401
                     -0.03367313 -0.08180340  0.00480114
                      0.07099302 -0.03914713 -0.00221035

 Rotated from first domain by 179.7 degrees about
 reciprocal axis  1.000  0.001 -0.032  and real axis  1.000  0.002 -0.010

 Twin law to convert hkl from first to      0.999   0.004  -0.019
 this domain (SHELXL TWIN matrix):          0.003  -1.000  -0.004
                                           -0.064   0.006  -0.999
```

CELL_NOW has identified an additional domain that fits 97.5% of the data with a tolerance of 0.2. The relationship between this domain and the first domain and the SHELXL TWIN matrix are reported. This example is for a rotational twin about the a* axis.

The output shows a suitable cell with its orientation matrix, figure of merit and the deviations of the input reflections from integer indices. This deviation is used to determine which reflections fit this cell and which are left for further searches for new domains.

11. Enter the maximum deviation from integer index cutoff (typically, the default is reasonable).

```
Maximum deviation from integer index [0.25]:
```

12. Enter the name of the .p4p file for this solution (in this example domain2.p4p).

```
.p4p or .spin file to write domain to:domain2.p4p


RLATT color-coding employed in file: domain2.p4p
 White: indexed for first domain
 Green: current domain (but not in a previous domain)
 Red: not yet indexed

   691 reflections within 0.250 of an integer index assigned to domain  2,
   119 of them exclusively;     3 reflections not yet assigned to a domain

 Re-refine initial cell (R), search for next domain (S), quit (Q) or choose
 new cell from list (enter number) [Q]
```

13. Enter <q> to exit CELL_NOW.

```
C:\struc\guest\twin>
```

NOTE: At this point CELL_NOW has indexed a two-component twin. The orientation matrices for the two components are in the domain2.p4p file.

10.5.2 CELL_NOW Output

After processing, in addition to the terminal output and the example._cn listing file written to disk, CELL_NOW writes two .p4p files, domain1.p4p and domain2.p4p. The second of these is a multicomponent .p4p for a two-component twin. It is shown below.

The following entries appear twice (once for each domain):

```
CELL, ORT1, ORT2, ORT3, ZEROES, ADCOR
CELL2, ORT12, ORT22, ORT32, ZEROES2, ADCOR2
```

This will eventually trigger SAINT to treat this data set as a twin.

```
FILEID SAINT        V6.43A        4.00        06/19/03 09:36:24 mraa
SITEID RufM                                 Ruf
TITLE  [RCpCrCl2.nTHF]n
CHEM   C40H58Cr2Cl4O2
CELL    12.6918   11.0161   14.6480   89.7011   90.9494   90.0010   2047.695
CELLSD   0.0025    0.0022    0.0029    0.0300    0.0300    0.0300     1.024
ORT1       0.001643376    -0.003574040    -0.068163186
ORT2      -0.034171116     0.081701167    -0.003850471
ORT3       0.070988722     0.039403379     0.000980361
ZEROS   0.0000000  0.3249754 -0.0041494    0.0000    0.0000    0.0000
ADCOR      2.9302     -0.0047     0.0003     -0.8959     0.0123     0.0937
CELL2   12.6918   11.0161   14.6480   89.7011   90.9494   90.0010   2047.695
CELLSD2  0.0025    0.0022    0.0029    0.0300    0.0300    0.0300     1.024
ORT12      0.005989663     0.004023272     0.068074010
ORT22     -0.033673126    -0.081803404     0.004801138
ORT32      0.070993021    -0.039147127    -0.002210349
ZEROS2  0.0000000  0.3249754 -0.0041494    0.0000    0.0000    0.0000
ADCOR2     2.9302     -0.0047     0.0003     -0.8959     0.0123     0.0937
SOURCE Mo     0.71073   0.70930   0.71359   2.00000   50.00   40.0
```

NOTE: SAINT+ users should be aware that APEX2 Version 2 can import .p4p files created with CELL_NOW, and the Integrate Images module will handle the multiple orientation matrices correctly. There is no longer a need to use the standalone SAINT+ program.

10.6 Configuring the Suite

The Bruker Suite includes the ability to customize many features of the program. Most customizations should be left to the site administrator, but some introduction is appropriate.

The following is a collection of examples of possible changes to the configuration files that control the look and feel of the GUI.

10.6.1 The bn-config Configuration File

The appearance and behavior of this Suite of programs can be modified by a configuration file. The programs read a series of configuration files in following order. As described below, a specification in a later file overwrites a setting in an earlier file.

For Windows: these files are (in order):

1. bn-config.py in the “SystemDrive” folder
2. bn-config.py in the “USERPROFILE” folder
3. bn-config in the current folder

For Linux/UNIX: these files are (in order):

1. /usr/local/lib/bn-config.py
2. .bn-config in your home directory (Please note the initial “.” character, which renders this file invisible for the normal `ls` command).
3. bn-config in your current directory.

If none of these files are present (e.g., when the program has just been installed), all parameters use built-in defaults. To change a single configuration parameter, create or edit one of the three files and put in a value for the parameter to be customized. You should:

- put a parameter in (1) if it should be the system default for all users;
- put it in (2) if it is your own default, and other people might want to use other values; and
- put it in (3) if it is only required for a single project.

The configuration files are read as Python programs, and therefore syntax is very important. The best way to use it is to only use variable assignments or function calls. If required, you can comment out lines from your configuration file by preceding them with a `#` character.

NOTE: Leading space is significant. Start all assignments in the first column. Variable names are case sensitive.

10.6.2 Global, User, and Sample-specific Variables

Configuration variables the system administrator wants to set globally for all users go into a file called “bn-config.py” that resides in:

- C:\ for Windows; or
- /usr/local/lib for Linux.

An individual user can set his or her own preferences for most configuration variables in a file in the home directory. This file is named:

- bn-config.py for Windows; or
- .bn-config for Linux.

NOTE: To find out where the user version of bn-config.py should be located (under Windows), enter `echo %UserProfile%` at a command prompt.

Additionally, configuration variables can be set for a specific sample using a file in the sample directory. For Windows and Linux, this file is named “bn-config”.

These files are read in the order given above, so values set globally are replaced by individual values and global and individual values are replaced by sample-specific values.

10.6.3 Configuration File Examples

Comments

A few notes about Python comments:

Three quotation marks in a row on a line (‟‟‟) start a comment section The next three quotation marks (‟‟‟) end the section.

The “pound” or “number sign” character (#)starts a simple comment. This can be anywhere on a line.

Default Color Scheme for Image Display

The default color scheme for the image display is given in:

```
colorramp='Colored_SCD.ICM'
```

This is the typical BAXS orange display.

The most frequently used other color choices are:

```
Black_On_White_SCD.ICM
White_On_Black_SCD.ICM
The_Blues_ANY.ICM
```

Other color ramps are in:

- Windows: C:\bn\src\gui\intensitycolormaps
- Linux: /usr/local/bn/src/gui/intensitycolormaps

User Positions

The detector distance is specified in millimeters. The goniometer angles are specified in degrees. To activate a user defined goniometer position, its userpos<#> variable and at least one of the positional variables must be uncommented. For instruments with a manual detector track, the detector distance variable (dx) should normally not be uncommented because of the risk of setting the distance to a different value than the actual distance. The chi angle setting must be uncommented for instruments with a Kappa goniostat.

An example user position:

```
userpos1 = "User Position 1"
userpos1_dx = 40.0
userpos1_2theta = 23.0
userpos1_omega = 23.0
userpos1_phi = 90.0
userpos1_chi = 54.74
```

CCD Detector Temperature Range

Acceptable CCD temperature:

```
ccdtemperaturerange=(-65,-55)
```

Automatically Connect to BIS

Automatically connect to the instrument when needed:

```
autoconnect=1
```

NOTE: This is a protected variable that is controlled by the System Administrator.

Custom Orientation Matrix Runs

The following five values define matrix runs:

```
perpendicular_matrix_runs_2theta=30
# Set the 2-theta for the matrix measurement:
perpendicular_matrix_runs_distance=41
# Set the detector distance for the matrix measurement:
perpendicular_matrix_runs_nr_sweeps=3
# Do three sweeps or runs:
perpendicular_matrix_runs_sweep=6
# Each run will be 6 degrees long.
perpendicular_matrix_runs_frametime=None
# The frame time is left to be the default.
```

Debug Messages for Scans

Set up to get some debug messages for scans. This is useful if the **Resume** command is failing:

```
debug=('goniometer.scan',)
debug=('expt.findresolution',)
```

Hardware Profile Used by Data Collection Strategy Module

Hardware profile used by the data collection strategy planner (also called COSMO; the hardware profile is generated by a program called COSMATIC):

```
bishost='bruker-server'
hardwareprofile='Kappa-APEX-II-euler.hrd'
#bishost='smart-apex'
#hardwareprofile='SMART-APEX-II.hrd'
```

Frames in a Network Shared Folder

IP address or DNS name of the file server used for storing images, followed by the name of a network shared folder on that server. Add additional path segments as comma-separated and quoted strings:

```
fileserver=('x8-client','frames')
```

To find out the DNS, enter the following at a command prompt:

- Windows: `echo %computername%`
- Linux: `hostname`

Supported Filename Protocols

Currently-supported filename protocols are `smb` for files that are accessed over a Microsoft Windows network, and `file` for local files:

```
universalfnprotocols=('smb','file')
```

10.6.4 Functionality Groups in the Configuration File

Sometimes a complete group of options need to be changed to get a specific functionality. For these cases, there are function calls that change a number of variables at once.

Due to the “default-like” nature of these function calls, they should probably be used only in the site-wide configuration files, and even there only at the beginning (but this is not enforced).

The available functions are:

- default_protein()

  This switches from the default “small-molecule” parameters to more protein-like parameters. At this time, the following parameters are set:

```
chiralonly=1
autochiralpointgroup=1
resomode=1
resolution=2.5
```

- default_noprotein()

  This sets the defaults back to small-molecule values.

10.6.5 Configuration File Variables

Table 10.3 — Configuration file variables

Variable	Type	Default Value	Description
anglemargin	floating point	1.0	Do not change.
autochiralpointgroup	boolean	0	This selects whether point groups are preferably chiral. This can be useful in a protein setting.
autoconnect	boolean	0	If this is set to 1, the connection to the BIS server will be created automatically without user intervention whenever required. This variable can only be changed in the system-wide configuration file.
axcrit	floating point angle	0.2 (degrees)	This is the criterion used by the automatic cell reduction algorithm to decide whether two axes coincide. Do not change.
badcolor	tuple of three integers	(255,190,190)	Color in the GUI that is associated with “bad” situations
busycolor	tuple of three integers	(240,240,96)	Color in the GUI that is associated with “busy” situations
bgdone	tuple of three integers	(96,240,96)	Color in the GUI that is associated with “done” situations
bgerror	tuple of three integers	(240,96,96)	Color in the GUI that is associated with “error” situations
bgimpossible	tuple of three integers	(240,240,96)	Color in the GUI that is associated with “impossible” situations
bgpossible	tuple of three integers	(96,240,96)	Color in the GUI that is associated with “possible” situations
bgtodo	tuple of three integers	(96,240,96)	Color in the GUI that is associated with “todo” situations
bgwarning	tuple of three integers	(240,240,96)	Color in the GUI that is associated with ’warning’ situations
bishost	string	localhost	The name or IP address of the BIS server computer
calibweaklevel	integer	10	Minimal I/sigma for peaks to be used in calibration routines.
ccdtemperaturerange	tuple of 2 integers	(-65,-35)	Acceptable range (minimal,maximal) of temperatures in °C for the detector. This is used only to indicate a red or a green status light for the CCD temperature in the status window.
chiralonly	boolean	0	If set to 1, only chiral point groups will be used at any moment. Do not modify.
colorramp	filename	’Colored_SCD.ICM’	Default color ramp used by the image display tool
communicationlog	filename	’communication.log’	File name used for logging all communication between application and BIS. If not set, no logfile will be kept.

Variable	Type	Default Value	Description
component1color	string (color name)	'green'	The "component#color" variables are obsolete. Use groupcolors instead.
component2color	string (color name)	'blue'	The "component#color" variables are obsolete. Use groupcolors instead.
component3color	string (color name)	'yellow'	The "component#color" variables are obsolete. Use groupcolors instead.
datcoldx	floating point	35.0	Default sample-to-detector distance for data collections.
dbtype	string	'postgresql'	Type of database engine used. Do not change.
dbhost	string	'127.0.0.1'	Name or IP address of the server running the database. The default is to run the database on the same host as the rest of the software, hence the default that refers to the local host.
dbport	string	'5432'	IP port to use to address the database. Normally dependent on database installation.
dbname	string	'BAXSdb'	Name of the database. Do not change.
debug	tuple of strings	()	This is a list of all modules that are to be debugged. This should not be touched, except if a developer asks you to add something here to help track a problem.
defaulthardwaretype	integer	4	If different hardware types are supported, this can be used to select which one should be used. 4=BIS. Do not touch.
detectorsizemargin	floating point	0.1	Do not change.
eventlogfile	filename	'bn-event.log'	File in which important events are logged during program runs.
fileserver	tuple of strings	()	Where experiment data files should be stored by default. First element in the tuple is the host name, second the share name, and the rest are successive folder names inside the share.
fileserverprotocol	string	'smb'	The protocol that should be used to address the file server. Should be either 'file' (local file) or 'smb' (network drive).
fontsize	integer	<none>	GUI font size in points
groupcolors	tuple of color names	('gray75','red','green','yellow','blue','magenta','cyan','white','gray25','brown','forest green','khaki','midnight blue','violet','turquoise','gray50')	

Variable	Type	Default Value	Description
guicolor	tuple of three integers making a color	0xd4,0xd0,0xc8	Overall background color of user interface.
guistyle	string	'windows'	The style of the GUI. Do not change.
hardwareprofile	filename	<none>	The hardware profile to be used by the Strategy Determination module (i.e., COSMO) for strategy calculations.
hashfilepattern	string	'_##_####'	Do not change.
headerdisplay	filename	<not set>	If present, this variable defines the name of a file containing the default image header items that should be displayed in the Image Display Area.
imageextension	string	'.sfrm'	Do not change.
imagemask	integer	0	Value of a mask used in viewing powder frames.
inhibitgc	boolean	0	Inhibits garbage collection. Do not change.
localfilemap	map	<none>	A map of network drives, translating local file names to equivalent network names. On Windows machines, this is set automatically. On UNIX, this should represent a copy of the relevant parts of the Samba configuration.
lowcurrent	floating point	5	Tube current in mA to set when an experiment finishes.
lowvoltage	floating point	20	Tube voltage in kV to set when an experiment finishes.
mailfrom	string	<not set>	The "From:" address of emails sent by the software (e.g., bug reports).
mailfromdomain	string	<not set>	The domain of the “From:” address of emails sent by the software. This will only be used if mailfrom is not set. The user name will be prepended.
matrixstrategy	list of scans	3 specific scans	The measurement strategy that is to be used to find a unit cell.
minimalpowerfraction	floating point	0.5	The minimal generator power as a fraction of the maximal power that will be considered sufficient to make a measurement.
mosaicity	floating point angle	0.4	Expected normal width of a reflection, in degrees.
nominalpowerfraction	floating point	0.9	The normal generator power as fraction of the maximal power that the generator should be set to before a measurement.
nominalvoltage	map	{'MO':60,'CU':45,'AG':60}	A definition of the nominal tube voltage in kV as a function of the target element.
orientdx	floating point	-5	The default crystal-to-detector distance for the “aligned images” module. If given as a negative number, it is counted from the rear of the DX sled.

Variable	Type	Default Value	Description
orientdegreetime	floating point	10.0	Default scan time in seconds per degree for an aligned image.
orientscanwidth	floating point	5.0	Default scan angle in degrees for an aligned image.
overlapcolor	color string	'red'	Color used for overlapping predicted spots
overlaycolor	color string	'green'	Color used for predicted spots
peakradiusfactor	floating point	2.2	How many times the single-sigma-spot radius should be used as integration radius. Do not change.
planes	string	'0kl h0l hk0'	List of precession planes that is suggested as default planes for the calculations.
planethickness	floating point	0.1	Default thickness of precession images
precessionmargin	integer	1	How many pixels at the outer edge of the detector are to be ignored in precession calculations.
prefereulerian	boolean	1	• 1 = prefer eulerian geometry over kappa geometry • 0 = prefer kappa geometry over eulerian geometry This should not be changed after installation.
preferreddirection	integer	-1	(default rotation direction of axes)
productline	string	'ANY'	A selection of the features in the program suite based on the type of application: • 'ANY' or 'ALL' = all products, • 'SCD' = single-crystal, • 'XRD' = powder, • 'XRF' = fluorescence
protectedvariables	map	{'protectedvariables':1,'autoconnect':1}	A selection of variable names and a limit to which configuration files can be used to change them. This can be used to make sure users do not overwrite system defaults, like is done by default for the autoconnect variable.
resolution	floating point	0.77	Default resolution to go for in an experiment. 0.77 is reasonable for standard small-molecule experiments.
scanrepr	integer	-1	Determines how scans are represented in text: • -1: scan angle from x to y length y-x • 0: scan angle length y-x starting from x • 1: scan angle from x to y
scanwidthtolerance	floating point angle	0.003	Tolerance in degrees that determines whether two scans are equal. Do not change.

Variable	Type	Default Value	Description
smbroot	string	'/smb'	UNIX only. Where Samba will mount shares made available from other hosts.
smtphost	string	'localhost'	Host of a machine that can be addressed via SMTP to send emails.
statuslines	integer	1000	Maximum number of lines displayed in the "Status" window.
temperaturerampspeed	integer	240	The speed to use for ramping the temperature of a low-temperature device, in K per hour.
universalfnprotocols	tuple of strings	('smb','file')	Allowed protocols to exchange files with BIS. Should be ('smb','file') when BIS is running on the same machine, and ('smb',) if BIS is running remotely.
userpos1	scan	<not set>	User-defined instrument axis position.
userpos2	scan	<not set>	User-defined instrument axis position.
userpos3	scan	<not set>	User-defined instrument axis position.
userpos4	scan	<not set>	User-defined instrument axis position.

Index

Copyright Notice Notices-ii

A

Abort command 2-9
abort data collection 5-28
Absorption correction
    and Face Indexing 10-7
Absorption Correction Type 6-16
Absorption Type 6-18
Access levels 3-1
Accounts 3-1
Acentric space groups
    and Intensity Statistics 6-22
Active Mask 6-8, 6-23
Align Crystal module 2-5
Anisotropic refinement 8-15
APEX2
    starting 3-2
APEX2 software 1-1
APEX2Server 2-5
Archive functions 2-8
Atom labelling
    in XSHELL 8-12
Atomic Displacement Parameters 8-16
AutoStructure 2-12
AUTOSTRUCTURE module 1-2, 2-12

B

bad runs
    omitting during scaling 6-20
Base Name 5-5
BCP
    required rights 3-1
BIS
    connecting to 3-2
    defined 2-3
    required for online mode 2-2
    starting 3-2
bn-config.py 10-38
Box tool 2-10
Bravais lattice 5-10, 5-11
    figure of merit 5-11
brukerinstrument.ini 2-4
brukersupport.com 1-5

C

Calibration module 2-12
Caution notice
    defined 1-4
Center Crystal module 2-11
Centric space groups
    and Intensity Statistics 6-22
Centroids 10-22
Change Password 2-8
Chiral compounds

specifying in XPREP 7-13
Chi-Squared plots 6-22
CIF file 8-21
Circle tool 2-10
Client
defined 2-2
Close sample 2-8
Compare Unit Cells 2-12
Compare Unit Cells module 2-12
Completeness 5-21
in XPREP 7-15
Connection... dialog 2-9
Constrain Metric Symmetry 6-6
Copy RES to INS 8-8
Correlate frames 5-6
Correlated frames 5-6
coverage statistics 6-13
Crystal Faces module 2-11, 10-7

D

D85 goniometer
centering the crystal 4-10
Damping Factor 6-6
Danger notice
defined 1-4
Data Collection Strategy module 2-11
data collection strategy modules 5-14
Describe module 2-11, 5-2
Determine Unit Cell
custom runs 5-4
Determine Unit Cell module 2-11, 5-3
Diagnostic plots 6-21
diagnostics
messages in BIS 2-3
Difference electron density maps 8-20
Diffraction Space Viewer module 2-12
Direct methods 8-6
Disconnect from BIS command 2-9
documentation revision numbers 1-5
Domains
in CELL_NOW 10-31
Doors (enclosure) 4-2
Dual-Space methods 8-6

E

EI button in XSHELL 8-12
Enable Box Size Refinement 6-6
Enable Orientation Refinement 6-6
Enable Periodic Refinement 6-6
Ewald sphere
and RMS XY 5-12
in refinement 5-12
Excluded shells 5-7
Execute button 5-26
Exit 2-8
Exiting during data collection 5-28
Experiment module 2-11
Export .p4p or .xml files 2-8

F

Face Indexing 2-11
Face List 10-9
Face normals 10-9
Factor g for initial weighting scheme 6-18
Find a Reflection 2-12
Find a Reflection module 2-12
Find Runs... button 6-3
First frame 2-10
Formula 8-4
in XPREP 7-16
frames
displaying 2-10
Frequency (Images) 6-6
Friedel pairs 5-16

G

Generate New Darks checkbox 5-28
Generate Report module 2-12

Generate Report with XCIF module 2-12
generator standby 10-3
Geometrically strained carbon atoms 8-18
Goniometer head
Huber 4-3
Nonius 4-3
unlocking sleds 4-1
Greek and Roman text 1-3
Groups
in Reciprocal Lattice Viewer 10-28
GUI 2-7

H

Help menu commands 2-9
High Resolution threshold 6-18
Histograms
in Scale module 6-19
in unit cell refinement 5-12
Hybridize All command 8-19
Hydrogen atoms
adding in XSHELL 8-18

I

I/sigma(I)
in unit cell determination 5-7, 5-9
ice resolution shells 5-7
icing 4-16
Image width 5-5
Import .p4p or .spin file 2-8
Import Runs from Experiment 6-4
Information on All Atoms 8-10, 8-16
Initial XYZ Box Size 6-6
Instrument menu commands 2-9
Integrate Images module 2-11
integrated intensities 6-1
integrating images from a CD or DVD 6-3
Intensity Statistics plots 6-22
Isolated reflections 5-9
Isotropic displacement parameters 8-16

J

JPEG output 8-23

K

KAPPA Series II goniometer
centering the crystal 4-4

L

Last frame 2-10
Least Squares 6-6
Line tool 2-10
Logging in 3-2
Login 2-8
Logout 2-8
ls button 6-13

M

Max. Number of Reflections 6-6
Mean I/sigma(I) threshold 6-18
Mean Weight 6-19
Menu Bar 2-8
Merge all equivalents
in XPREP 7-15
Microscope 4-1
module
defined 2-4
Mosaicity 6-9
mosaicity 5-3
Mount button 4-2
Movie view 2-10
Movie view for frames 2-10
Movie view rate slider 2-10

N

New Sample 2-8, 5-2
New sample 2-10
Next frame 2-10
Next run 2-10
No. of Refinement Cycles 6-18

Normals
in Face Indexing 10-9
North American service center 1-7
Note
defined 1-4
Numerical Absorption Correction (From Face Indices) 10-14

O

Open frame 2-10
Open in XP 8-8
Open in XSHELL 8-8
Open sample 2-8, 2-10
OpenGL rendering alert 8-23
Oriented Scans module 2-11

P

p4p file
writing from Reciprocal Lattice Viewer 10-30
P4P files
CELL_NOW output 10-36
Patterson methods 8-6
pink fields (invalid entries) 2-7
Planarity
and MPLA command 8-22
Play Tic Tac Toe module 2-12
Plotting lines
drawing 2-10
poor-quality crystals 4-21
Precession Images 2-12
Precession Images module 2-12, 7-1, 7-18
Previous frame 2-10
Previous run 2-10
Publication
Generate ACTA(CIF) Information File 8-21
refinement in XSHELL 8-21

Q

Q-peaks (unassigned peaks) 8-5, 8-11, 8-18

R

R(int)
variations 6-21
vs. resolution 6-22
R1 value 8-14, 8-15, 8-20
Radiation Safety Officer 1-6
Reciprocal Lattice Viewer module 2-11, 10-15
Redundancy
in XPREP 7-15
referenced documentation 1-5
Refine
with XL 8-8
Refine button 6-19
Refinement Parameters 6-6
Reflection Group Combo Box 5-8
Relabelling peaks 8-13
Reopen sample 2-8
Resolution circle
drawing 2-10
Resolution Limit 6-2
Restraints esd for Scale Factors 6-18
reverse Movie view 2-10
reverse-beam geometry 5-16
Rint 7-15
RLATT 2-11
RMS XY 5-12
Rshell values in SAINT output 6-13
Rsigma 7-15
Rsym
overall 6-13
Run command 2-8
R-values 6-19

S

SADABS 2-1
safety issues 1-6
SAINT 2-1
SAINT file format description 6-1

SaintChart 6-10
Sample Database
automatic startup and shutdown 2-6
default account 3-2
defined 2-6
Sample menu commands 2-8
Sample Notes window 2-8, 2-10
Save sample 2-8, 2-10
Scale module 2-11
closing 6-23
Scale Variations plot 6-21
scaling 6-1
Server
defined 2-2
Simple Scans module 2-5, 2-11
Smooth images 5-7
Sort Runs for Completeness 5-23
Space Group Determination module 2-12, 7-1
Space Groups and Statistics module 2-12
Spatial Distribution plots 6-23
Stack Bar view 2-13
Status window 2-9
Stop button 2-10
stop Movie view 2-10
Strategy module 2-11
Strategy view (detailed) 5-24
Structure refinement
module 2-12
overview 8-13
Structure Refinement module 2-12, 8-7
Structure Solution module 2-12, 8-4
Suite
rights required to run 3-1
Supercell problems
in CELL_NOW 10-34
Symmetry
higher symmetry cell search with XPREP 7-12
System Calibration module 2-12
Systematic absences
and XPREP 7-14

T

Task Bar 2-11
Task Display Area 2-14
technical support 1-7
Thermal Ellipsoids
plots 8-22
Tic Tac Toe 2-12
Title Bar 2-8
Toggle Shutter command 2-9
Tolerance
in refinement 5-12
Tool Icon Bar 2-10
Torsion angles
and CONF command 8-22
Transform Unit Cell module 2-11
Tree View 2-13
T-tool (in Crystal Faces module) 10-9
Twinned crystals
and precession images 7-20
Typographical conventions 1-3

U

unconstrained unit cell refinement 6-14
Unit Cell Combo Box 5-12
Unit Cell tool
in Reciprocal Lattice Viewer 10-27
Unwarp and Convert Images module 2-12
user positions 10-39

V

Validate button 5-28
Variables in bn-config.py 10-38
Video zip stream 10-7
View Images 2-11
View Images module 2-11
View Molecule module 2-12
vzs file 10-8

W

Water ice resolution shells 5-7
What's This? Help function 1-7, 2-10
Whole reflections 5-9
Windows menu commands 2-9
work directory 6-1

X

XL 2-1
XM 2-1
XPREP 2-1
X-ray safety 1-6
XRD² Eval module 2-12
XRD2 Eval module 1-2
XS 2-1
XSHELL
changing displayed atom sizes 8-22

Y

ylid 1-2

Z

Zvalues 7-16